DETAILED SPACIAL AND TIME RESOLVED STUDIES OF LUBRICANT DEGRADATION IN MULTI-CYLINDER DIESEL ENGINES

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ABSTRACT

This work formed part of the North American Space Agency /US Department of Energy project to produce a 55% thermodynamic efficient diesel engine, which required the construction of a "low heat rejection" engine. Work was carried out on a Caterpillar 3406B, a commercial, multi-cylinder, 'low emission' diesel engine.

The work evaluated oil mass and gas flow, the chemical and physical degradation of commercial and experimental lubricants, the effects the sulphur content in diesel fuels had on the particulate size in lubricants, lubricant transport through the piston ring pack of the Caterpillar 3406B, and the effects different piston ring packs had on both lubricant degradation and lubricant transport.

Piston ring zone sampling was used throughout this work. Lubricant samples were taken from four positions on the Caterpillar 3406B piston, namely the 2nd compression ring, accumulator groove, 1st compression ring and base of the crown land. A duel sampling system was developed which enabled lubricant samples to be taken from two sampling positions on the piston simultaneously. This sampling system proved to be reliable with over 650 hours of operation, at various engine speeds and loads.

Oil mass and gas flow experiments showed complex behaviour. Oil mass and gas flow generally increased as engine speed and load increased, however 'peaks' of oil mass and gas flow were observed at various engine speed and load combinations, which may be a reflection of the piston ring pack design. Examination of oil mass obtained from 1991 and 1994 US Emission control piston ring packs, under conditions of low engine speed and low load, demonstrated that the 1994 piston rings allowed more lubricant to enter the ring pack. Significantly less oil was obtained from the base of the crown land with the 1994 piston ring pack, highlighting their superiority with regards to reducing lubricant related emissions.

Measurement of the chemical and physical degradation of various lubricants demonstrated that degradation increased as the sampling position moved closer to the piston crown. Different degradation levels were noted whether the sampling position was on the piston face or behind the piston rings. Analysis of lubricant degradation using statistical techniques demonstrated that

lubricant type, sampling position, fuel sulphur content and piston ring pack significantly affected lubricant degradation. The work showed that engine speed and load did not effect lubricant degradation. The use of statistical analysis demonstrated that lubricant samples could be graded according to their level of degradation, which highlights the potential use of piston ring zone sampling as a quick screening method for evaluating experimental lubricants.

Lowering the sulphur content in diesel fuels had the effect of increasing the size of particulates in the lubricant. This questions the effectiveness of the dispersant additives in the lubricant formulation, and highlights the need for more effective dispersant additives to be used with lower sulphur diesel fuel.

Lubricant transport to the 1st compression ring was found to be considerably slower in a modern engine, when compared to older engines. This demonstrates that there may be a need for engine manufacturers to recommend the use of synthetic lubricants over the use of mineral based lubricants. A lubricant flow model was developed which showed that the piston rings act as secondary oil control rings and that 19% of the lubricant that reaches the 2nd compression ring would be transported to the base of the crown land.

Comparison of lubricant degradation in the 1991 and 1994 US Emission control piston ring packs demonstrated that the 1994 piston ring pack degraded lubricants to a lesser extent, which is thought to be due to there being more oil present in this piston ring pack. The lubricant transport measurements showed that the transport time to the 1st compression ring was less for the 1994 piston rings than the 1991 piston rings, and that the 1994 rings allowed more lubricant to be passed from the 2nd compression ring to the 1st compression ring.

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CHAPTER 1

Introduction

CHAPTER 1 Introduction

1.1 Project Rationale

This study was initiated by Professors M.F. Fox and D.J. Picken of De Montfort University and Franklin Kelley of Caterpillar Inc. Peoria, USA. Sponsorship has been provided by the North American Space Agency (NASA)/US Department of Energy (DoE) through Caterpillar INC, Peoria, Illinois, USA. Technical help, fuels and lubricants have been provided by BP Amoco, formally BP Research and Engineering Centre, Sunbury-on-Thames, UK and Castrol Research Laboratories, Pangborne, UK.

Previous piston ring zone work has been carried out at De Montfort University on single cylinder petrol[1.1] and diesel engines[1.2]. This project involved work on a commercial, low emission, six cylinder Caterpillar 3406B diesel engine, modified to undertake piston ring zone work (Contract number DEN 3-374). The work carried out at De Montfort University was part of the NASA/US DoE project to produce a 55% thermodynamic efficient diesel engine[1.3], which required the construction of a "low heat rejection" engine. The production of a "low heat rejection" engine could, potentially, lead to problems of rapid oil degradation due to the engine being run at an elevated temperature, when compared to conventional engines.

The work carried out at De Montfort University was to evaluate the chemical and physical degradation of commercial and experimental lubricants in a Caterpillar 3406B multi-cylinder diesel engine by extracting lubricant from the piston ring zone, evaluate lubricant transport through the piston ring pack of the Caterpillar 3406B, and evaluate the effects different piston ring packs have on both lubricant degradation and lubricant transport.

The rationale behind the project was:

- The current lack of understanding of how lubricants degrade in the piston ring area, which is becoming more important as new government regulations[1.4] require lower emissions. As combustion related emissions have decreased the emissions arising from lubricants have become more important, and need to be reduced. To comply with these regulations new piston ring packs have been designed which reduce the amount of lubricating oil in the piston

ring area, therefore the lubricants have to perform under more arduous conditions with much thinner oil films. This can lead to problems of physical and chemical breakdown of the lubricant.

- Provide understanding of lubricant transport in the piston ring zone, via experimentation, and how this transport is affected by piston ring pack design.
- The length of time it takes to test lubricants by conventional sump testing.
- The limited amount of information that has previously been gained by the complex nature of the results obtained from analytical testing of formulated lubricants.

The work carried out in this study has been reported in six chapters. Chapter 1 outlines the aims and objectives of the project, introduces the concepts of lubrication, friction, piston ring packs, and describes the advantages and disadvantages associated with sump and piston ring zone lubricant test methods. Chapter 2 describes the piston ring zone sampling technique employed, the engine and analytical test methods performed during the study, examines gas flow and lubricant mass obtained from the piston ring zone sampling positions, and reports the preliminary results obtained with respect to lubricant degradation and lubricant transport. Chapter 3 describes in detail the lubricant transport experiments and findings, in relation to lubricant transport from the sump to the piston ring zone sampling positions. Chapter 4 contains a statistical analysis of the analytical data obtained from the study, in relation to lubricant degradation as a factor of engine speed, oil type, fuel sulphur content, piston ring zone sampling position and piston ring pack type. Chapter 5 examines the effects of piston ring pack design on lubricant mass in the piston ring zone and on lubricant transport to the piston ring zone. Chapter 6 contains conclusions from the study and recommendations for further work.

1.2 Project Aims and Objectives

The main aims of this work were to investigate the relative degradation of different formulations of lubricants from various sampling positions in the piston ring zone of a commercial multi-cylinder diesel engine, investigate lubricant transport in the piston ring zone and investigate the effects of piston ring pack design on lubricant degradation and lubricant transport. The objectives can therefore be split into the following areas of work:

1. Development and installation of a piston ring zone sampling system in the Caterpillar 3406B engine. The size of the piston meant oil could be sampled from four positions, behind the 2nd ring, between the 1st and 2nd ring (accumulator groove), behind the 1st ring and above the 1st ring (base of the crown land).
2. Running the Caterpillar 3406B engine at differing speeds and loads to obtain oil samples from each sampling position, using various commercial and experimental oil formulations. The lubricants used ranged from standard quality unmodified mineral oil to very good quality synthetic oil, therefore lubricant degradation could be related to differences in oil formulations with respect to sample position, engine speed and load.
3. Analytical testing of lubricant samples by industry standard test methods to investigate lubricant degradation, in order to distinguish between the different base oil stock and additive packs of the lubricants under test.
4. Investigation of the particle size distribution of insoluble contents within the lubricant samples, in order to identify how this was affected by sampling position, lubricant type and fuel sulphur content.
5. Measurement of oil transfer from the sump to the four sampling positions on the piston as a function of time, in order to develop a model of lubricant transport through the piston ring pack.
6. Investigation of the effects of piston ring pack design on lubricant degradation and lubricant transport.

1.3 Introduction to Lubrication

1.3.1 A Brief History of Lubrication

Lubrication in one form or another has been in use for many years[1.5]. The ancient Egyptians used tallow (animal fat) to lubricate the axles of their carts and chariots. This became known as boundary lubrication, where load is carried by a thin layer of lubricant. The advent of mineral oils made lubrication more reliable because of their enhanced film strength properties, which meant longer lasting lubrication. The invention of the steam engine brought forth a previously unexploited technique in lubrication, where bearings were no longer in occasional contact with one another as in boundary lubrication, but separated by a fluid film thick enough to prevent any metal-to-metal contact. To achieve this thin film the bearing's geometry had to be such that a 'wedge' of lubricant was produced in the travelling direction of the bearing, know as hydrodynamic lubrication. Synthetically produced lubricants are becoming more common today, and although more costly than mineral oils, they are more resistant to degradation, so have longer working lives.

A variety of materials can be classified as lubricants, either solids, liquids or gases. However the main job of the lubricant is the same, to assist the sliding of two surfaces when the lubricant is placed between them.

1.3.2 The Basic Requirements of a Lubricant

When solid surfaces are in sliding contact, friction and wear can occur. Friction is a mechanical resistance to the movement of the surfaces, leading to generation of heat, which wastes power. Wear occurs when two surfaces pass over one another. This leads to the loss of mechanical conformity, which in turn reduces the lifetime of the mechanism.

Both friction and wear are undesirable, but they can be almost eliminated if the surfaces are prevented from touching. This is the essential purpose of any lubricant.

A lubricant has to provide chemical and physical protection of the surfaces which it comes into contact with and prevent corrosion by atmospheric gases, water vapour and other hostile agents.

The main functions of lubricants are shown below:

- Reduce friction and wear.
- Transfer heat away from surfaces.
- Disperse contaminants.
- Protect against corrosion.
- Provide good seals for engine parts.
- Resist the tendency of heat and oxygen to change the oil's properties.

1.3.3 Friction

All surfaces are uneven if they are magnified to a sufficient scale. When two surfaces come into contact they will only touch at the high spots, known as asperities, therefore the asperities are the points where the loads are supported and frictional forces are generated.
Leonardo Da Vinci developed the two fundamental laws of friction, shown below:

1. The frictional force is independent of the area of contact
2. The frictional force is proportional to the load applied

From the 2nd law the coefficient of friction, μ, is derived.

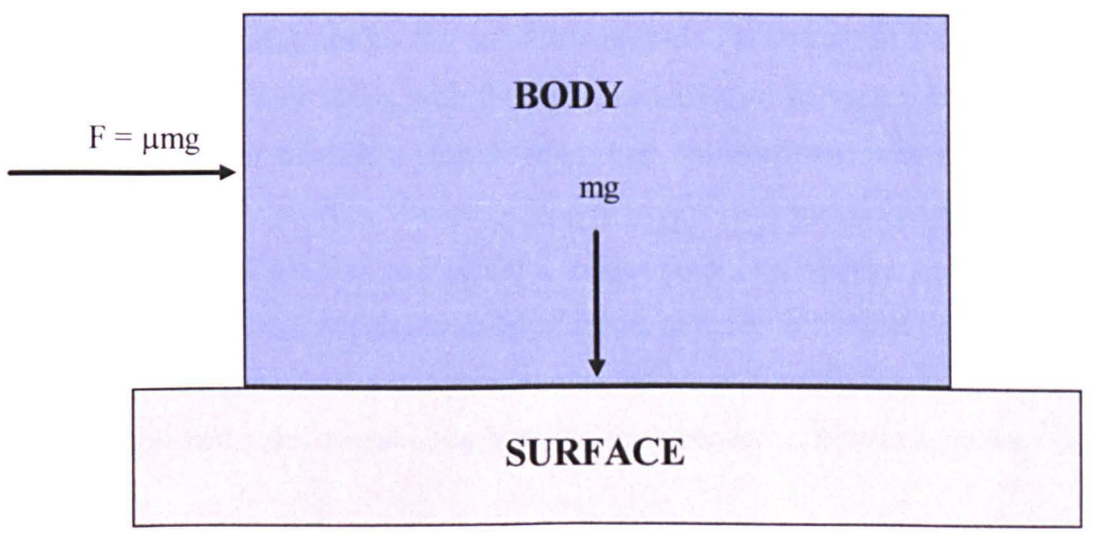

Fig. 1.1 Diagram Representing the Theory of Sliding Friction

Fig. 1.1 represents a body of load m resting on a level surface, under the effects of gravity g. Lateral force needs to be applied if the friction between the body and the surface is to be overcome. If F is the force required to balance the resting force of the friction, then any increase in the lateral force will cause sliding. Thus the friction equation is produced, which is shown in the equation below.

$\mathbf{F = \mu mg}$ where F is Force, μ is the coefficient of friction, m is mass and g is gravity

If the horizontal surface is covered in lubricant then μ is reduced and becomes independent of the contacting surfaces. However, μ is dependent on the nature of the lubricant. If m is small (i.e. low mass) the mass will float on the surface and when the mass is moved it will remain parallel to the horizontal surface. However if m is large (i.e. large mass) then the block will sink into the lubricant. Once the block is moved an oil wedge of lubricant will build up, which is known as hydrodynamic lubrication.

1.3.4 Lubricant Additives

If a pure base oil was used in an engine then it would probably not last more than a few weeks. The oil would have changed to such an extent that most of the beneficial properties would have been lost. If, however, small amounts of different types of additives are introduced, the lifespan is greatly increased. Additives increase the lifespan of the oil by combating the extreme physical stress and chemical degradation that an oil is subjected to in an internal combustion engine. Oil additive chemistry is complex, with the additives having to perform many functions without interacting with one another; a process which may produce even more harmful contaminants than the combustion process. Several additives have to be blended into the oil and remain in stable suspension or solution, as it can be a serious problem if additives drop out of suspension i.e. piston ring sticking brought about by additives adhering to metallic surfaces. The additives used in the oil may not be the best possible chemicals at performing the desired task, but are the most suitable under the overall conditions explained above. Lubricant additives fall into three categories:

1. Those that modify the characteristics of the base oil, thus making the oil more suitable for internal combustion engines.

internal combustion engines.

2. Those that protect the oil from physical stress and chemical degradation.
3. Those that protect the engine from the combustion process and the contaminants that may be formed by that process.

Table 1.1 gives a brief summary of the additives commonly found in lubricants, and the functions they perform.

Example of Additive	Action on Lubricant
Polymers	Viscosity index improvers
Polymethacrylate esters	Pour point depressants
Zinc Dialkydithiophosphates	Antioxidants, anti-wear, corrosion inhibitors
Organic amines	Metal deactivators
Silicones	Anti-foaming agent
Dibenzyl disulphide	Extreme Pressure additive
Calcium/Magnesium Sulphonate	Detergents
Alkylsuccinamide	Dispersants

Table 1.1 Common Lubricant Additives and the Functions they Perform

1.4 Piston Ring Packs and Lubrication Regime

Piston rings control lubrication between the piston and the cylinder liner[1.6]. The two types of piston rings (compression and oil control) perform the following tasks:

1. Provide a seal between the piston and cylinder liner to retain gas pressure and prevent blow-by.
2. Provide and control adequate amounts of lubricant to the cylinder liner to sustain high thrust and gas force and at the same time keep oil consumption to a minimum.
3. Control piston temperature by assisting in the heat transfer from the piston to the cylinder liner.

The design of the piston rings in the Caterpillar 3406B are a double 'knife edge' oil control ring, a stepped 2nd compression ring and a keystone 1st compression ring. These piston rings are shown schematically in Fig 1.2.

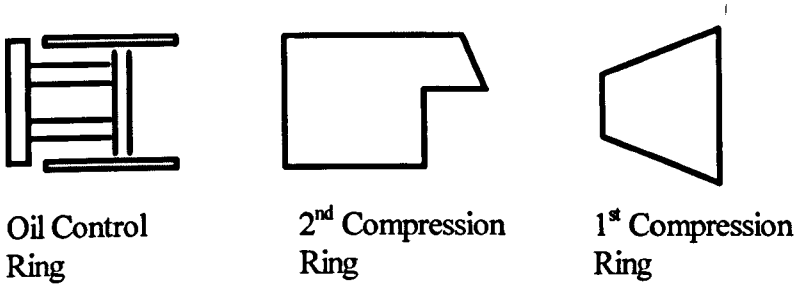

Fig. 1.2 Schematic Representation of the Piston Rings in the Caterpillar 3406B

Piston rings are designed to facilitate the formation of hydrodynamic lubricant oil films. The 1st compression ring is designed to allow the smallest possible amount of lubricant to enter the combustion chamber, and reduce blow by gases entering the crankcase. The 2nd compression ring is designed to reduce the pressure drop across the 1st compression ring, whilst controlling the oil flow to the top ring. The oil control ring controls the amount of lubricant entering the piston ring zone by distributing oil onto the cylinder liner evenly, and returning excess oil to the crankcase.

The tension in all the piston rings holds them against the cylinder liner, and hence contributes to friction. The gas pressure generated by the combustion process also 'forces' the piston rings against the wall of the cylinder liner, thus increasing friction.

The lubricant regime in the piston ring zone is complex. Piston rings are designed to produce a hydrodynamic lubrication regime. However, due to changes in the forces applied to the rings during the combustion cycle and changes in lubricant temperature effecting lubricant film thickness, the lubrication regime quickly changes from thick-film hydrodynamic to boundary lubrication.

Due to the changes in lubricant regimes in a very short time period the need for a 'good quality' lubricant is highlighted, therefore carefully designed lubricants are needed in the piston ring zone.

It should be noted that changes to Government legislation to reduce emissions and the need to produce 'low heat rejection' engines to improve efficiency , will increase the arduous conditions in the piston ring zone, and increase the need for the development and use for even 'better quality' lubricants.

1.5 Comparison of Advantages and Disadvantages of Sump and Piston Ring Zone Sampling

This work evolves the removal of oil from the piston ring zone of an operating diesel engine. Piston ring zone sampling is a novel way of sampling oil, compared to standard sump testing. There are however, advantages and disadvantages in using this method.

1.5.1 Advantages of Sump Sampling

1. A large oil volume is obtained, therefore many analytical tests can be carried out and repeated, as necessary.
2. The system is reliable. Only engine or oil failure will stop the test.
3. Sump testing is the industry standard lubricant test, with many documented tests undertaken.

1.5.2 Disadvantages of Sump Sampling

1. 100+ hours must elapse before the oil samples are available to test, therefore the whole test process is slow.
2. The cost of running an engine for 100+ hours.
3. It may not be possible to run all potential oil formulations available due to cost and time factors, therefore valuable data about oil formulations and how they perform will not be known.

1.5.3 Advantages of Ring Zone Sampling

1. Drastically reduced engine run times, but the same degree of oil degradation obtained.
2. Reduced cost of running the engine in terms of fuel and technician time.
3. An earlier indication as to whether an oil is going to pass the test. It is possible to know within 1 day whether the oil is acceptable, compared to at least 4-5 days with sump sampling tests.

1.5.4 Disadvantages of Piston Ring Zone Sampling

1) The small volume of sample obtained means that some conventional analytical tests can not be carried out, therefore new methods of analysis or new equipment must be developed to cope with the reduced volume of sample. Also, the small sample volume makes repeat testing difficult.

2) A skilled technician is needed to build and monitor the sampling system.

3) The system can be unreliable, with breakages happening fairly frequently. This results in the engine being stopped in order to dismantle and replace of the sampling system.

4) The sample is generally more contaminated with fuel and water than sump samples. These contaminates can make analysis difficult. However, these samples do represent the lubricant regime in the piston ring zone .

5) Piston Ring zone sampling is not a recognised test method as yet, with no proper documentation.

Subsequent chapters will discuss in detail the piston ring zone work carried out on the Caterpillar 3406B.

CHAPTER 2

Experimental Methods and Preliminary Results

CHAPTER 2 Experimental Methods and Preliminary Results

This Chapter sets out the aims, methods and analyses (chemical, mechanical and numerical) for this work. It explains in detail the techniques and methods used, so that the results of subsequent Chapter's research areas can be described and expanded in a more fundamental manner with out re-iterating those techniques and methodologies.

2.1 Aims of the Research Project

2.1.1 General Programme Aims

This work has formed part of the US Department of Energy/NASA programme[2.1] to develop a 'Low Heat Rejection' (LHR) diesel engine. The LHR diesel engine offers the possibility of an increased thermodynamic cycle efficiency through the use of advanced materials and higher engine operating temperatures. Materials used include ceramic Thermal Barrier Coated (TBC) pistons[2.2], ceramic coated intake and exhaust ports[2.3] and zirconia cylinder head inserts[2.4], together with turbo-compounding. The overall research objective is to produce a 55% thermodynamic cycle efficiency, equivalent to 512 grams Class A fuel kW^{-1}. This translates into a 40,000 kg payload hauled at 88 $kmhr^{-1}$ with a consumption of 23.6 ℓ 100 km^{-1}.

2.1.2 Specific Aims of this Project

The aims of this work were to investigate the lubricant requirements for the LHR engine, described by Waltz[2.5], under US Research Contract DEN-3-374. The aim of this research can be set out as follows;

'to find a reliable method of investigating lubricating oils in a medium speed multi-cylinder engine, so that lubricant degradation can be monitored and measured.'

Objectives:

- to install a commercial multi-cylinder diesel engine and modify the engine so that ring zone sampling can be achieved,
- design and develop a reliable sampling system to obtain lubricant samples from the piston ring zone,
- develop the sampling system so that lubricant can be sampled from more than one piston ring pack position simultaneously,
- measure oil and gas flow in the piston ring zone under conditions of varying engine speed and load,
- measure lubricant degradation as a function of sampling position, engine speed and engine load with lubricants of differing quality (i.e. 'standard' quality mineral base stock through to 'very good' quality synthetic lubricant), using analytical techniques developed,
- investigate particle size distribution in lubricant samples obtained as a function of sampling position and diesel fuel sulphur content,
- measurement of lubricant transport from the sump to the sampling positions, using suitable marker techniques,
- measure the temperature profile of the sampling piston using 'Templugs^{R}', and relate these results to oil consumption,
- develop models of oil transport through the ring pack.
- investigate the effect of 1991 and 1994 US Emission control ring packs with respect to lubricant degradation and lubricant transport.

2.2 Test Engine Installation Components

The major components of the test engine installation were:

- the engine test bed and concrete base,
- 3406B Caterpillar engine,
- Froude DPX4 Dynamometer,
- Fenner flexible flywheel coupling,
- cooling system.

2.2.1 Engine Test Bed and Concrete Base

To provide a strong mounting for the test bed and engine a concrete base, strengthened by steel mesh, measuring 2.75m by 1.8m by 0.9m deep was built in the floor of the engine test laboratory. The periphery of the concrete base was lined with cork to dampen vibration.

The test bed was manufactured from 0.25m by 0.125m 'I' section mild steel beams. Matting was fixed between the test bed and concrete block to take up any surface irregularities. Floor holding bolts fixed the test bed to the concrete block. The test bed was designed to accommodate the industrial mounting brackets on the engine, the flywheel coupling length and the mounting holes on the DPX4 dynamometer.

2.2.2 Test Engine (Caterpillar 3406B)

The engine used for this work was a Caterpillar 3406B, a commercial, low emission, turbocharged, in-line six cylinder, 14.5 litre diesel engine. Commercial uses for this engine are in the fields of industrial, marine and transport propulsion. The engine has a bore/stroke of 137mm by 165mm respectively. Fuel is supplied to the engine by direct injection to produce 325 BHP (242 kW) at full load speed of 2100 rev min^{-1}. The engine has a wiring loom incorporating safety features to prevent damage by over speeding, high cooling water temperature and low oil pressure. The engine was fitted with pre-1991 US emission control piston ring packs. The engine was run on 1% sulphur reference diesel fuel, later work used lower sulphur diesel fuel.

The engine was modified to undertake ring zone sampling by machining a rectangular hole in the crankcase at the fifth cylinder position. An aluminium box was constructed to fit over the rectangular hole.

2.2.3 Froude DPX4 Dynamometer

The power produced by the engine was absorbed by a Heenan and Froude DPX4 dynamometer, which converted the engine power into waste heat as warm water. This water was fed into a heat exchanger to cool the water from the engine cooling system. The power absorbing capacity of the DPX4 dynamometer is approximately 350 BHP (261 kW) at 2000 rev min^{-1}. This was measured by means of weights suspended from a balance arm, in conjunction with a spring balance to indicate small variations in load. The dynamometer was supplied by mains water. The load applied to the engine was measured by the weight suspended from the balance arm and the engine speed.

The weight applied load was converted into BHP by the following calculation;

$$BHP = \frac{WN}{2400}$$

Where:-W = applied load (lbs)

N = Engine speed (rev min^{-1})

BHP was converted to the SI unit of load, kW, by using the following calculation:-

$$1\ HP = 0.746\ kW$$

Table 2.1 shows typical loads applied to engine speeds from 750 to 1800 rev min^{-1}, for the Caterpillar 3406B.

Engine Speed (rev min^{-1})	Applied Load (BHP)	Applied Load (kW)
750	89	66
1200	140	104
1500	175	131
1800	265	198

Table 2.1 Typical Engine Loads Applied at Differing Engine Speeds

2.2.4 Flexible Flywheel Coupling

A flexible coupling, with the advantages of cost, compactness and inherent damping qualities connected the engine to the dynamometer. The choice of the coupling was dependent on such variables as, the maximum power and rev min^{-1} of the engine, the maximum torque produced and the rated stiffness of the flexible element. The Fenner coupling chosen was a standard item, apart from a special adapter machined to fit the coupling to the taper shaft of the dynamometer.

2.2.5 Cooling System

The cooling system consisted of an open system for the dynamometer and heat exchanger, which was external to the engine, and a closed system for the engine and the heat exchanger, which was internal to the engine. The open system consisted of a mains water supply to the inlet on the dynamometer. The outlet water from the dynamometer was then fed into the inlet cooling tubestack of the heat exchanger, which cooled the circulating water from the engine cooling system. The cooling water was then fed to a drain. Connections between the mains feed, dynamometer, heat exchanger and the drain were reinforced plastic tubing to minimise flow resistance. Thermocouples were positioned in the system to monitored the temperature of the inlet water to the dynamometer, the outlet from the dynamometer to the heat exchanger inlet and the heat exchanger outlet to the drain, Fig 2.1. During the research programme the thermocouples were repositioned at various times to measure the water inlet to the cylinder head, the cylinder head outlet to the oil cooler and the oil temperature at the cylinder oil jets.

2.3 Installation

The hole positions on the engines industrial mountings were checked against the installation drawings for the 3406B and found to agree. The holes were then marked out and checked by triangulation to avoid the engine being mounted out of square. The holes were then drilled and checked for accuracy. The engine was positioned on the test bed using a 1.5 tonne crane and then bolted to the test bed. Using the crane the dynamometer was

positioned on the test bed, and the coupling carefully connected to pull the dynamometer into position. The dynamometer was aligned to within specifications and then tightened into place. The Fenner flexible coupling specification allowed a maximum tolerance of 25μm between shaft centre lines. This installation attained a difference of 3-4μm, measured by a clock gauge. The test engine installation, including power absorption and cooling system, can be seen in Fig 2.1.

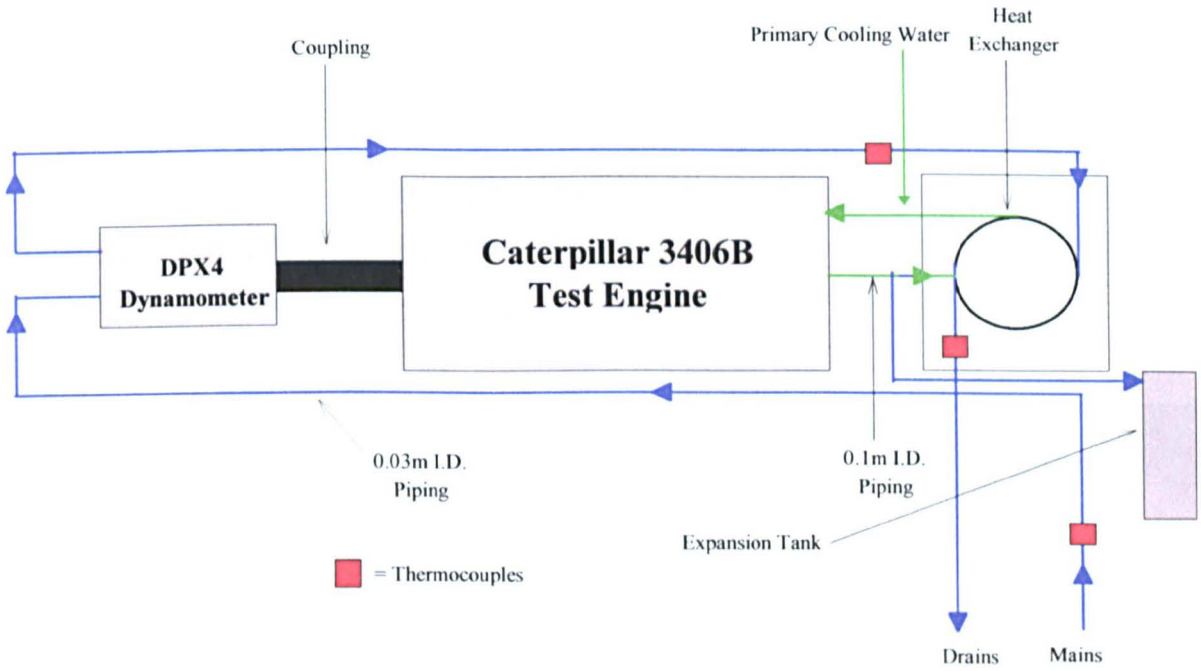

Fig. 2.1 Power Absorption and Cooling System for 3406B Caterpillar Engine

Photographs of the Caterpillar 3406B are shown in Appendix A.

2.4 Sampling Positions on the Caterpillar 3406B Piston

Previous work on piston ring zone sampling has been carried out at De Montfort University on single cylinder diesel[2.6] and single cylinder petrol engines[2.7]. These techniques involved removing lubricant from one position, the Top Ring Zone (TRZ). Due to the size of the Caterpillar piston (diameter 137mm) more than one sampling position on the piston was feasible. After careful consideration it was decided that lubricant could be taken from four

positions on the piston, using previous TRZ sampling systems as a guide. Table 2.2 shows these sampling positions and their relative distances from the edge of the piston crown.

Two pistons were modified to accommodate the sampling positions, one piston to sample from behind the 2nd and 1st rings (Position 1 and Position 3) and one to sample on the face of the piston, at the accumulator groove and base of the crown land, (Position 2 and Position 4). In each case the piston had two 1mm holes drilled, at 180° from one another, from the sampling point into the piston. An oil way was drilled and tapped inside the piston, so that a stainless steel insert could be fitted.

The inserts were standard High Performance Liquid Chromatography (HPLC) fittings, fitted with a short piece of HPLC thick wall steel tubing. Fig. 2.2 shows a schematic diagram of the sampling positions and the lubricant flow regime.

Sampling Position	Designated Sampling Position Number	Distance from Edge of Piston Crown / mm
Behind 2nd ring	1	33
In-between 2nd and 1st Ring (Accumulator Groove)	2	29
Behind 1st Ring	3	23
Above 1st Ring (Base of the Crown Land)	4	18

Table 2.2 Sampling Positions Chosen and the Distance From Piston Crown

2.5 Development of the Ring Zone Sampling System for the Caterpillar 3406B

Piston ring zone sampling has been undertaken previously at De Montfort University, section 2.4, using various single cylinder diesel and petrol engines. These sampling systems were based, to a greater or lesser degree, on lightweight, flexible, PTFE tubing with some stainless steel, micro-bore, tubing inserted at strategic points in the system. All were

designed to obtain lubricant samples from behind the top piston ring, at various engine speeds and loads, leading outside the engine into collector vessels. Because of the limited space within the engines, sampling could only be achieved from one point at a time. Comparative samples from other rings/points involved dismantling the engine and connecting the system to the next point. True comparisons of samples could be affected by the change over and possible differences in the operating environment.

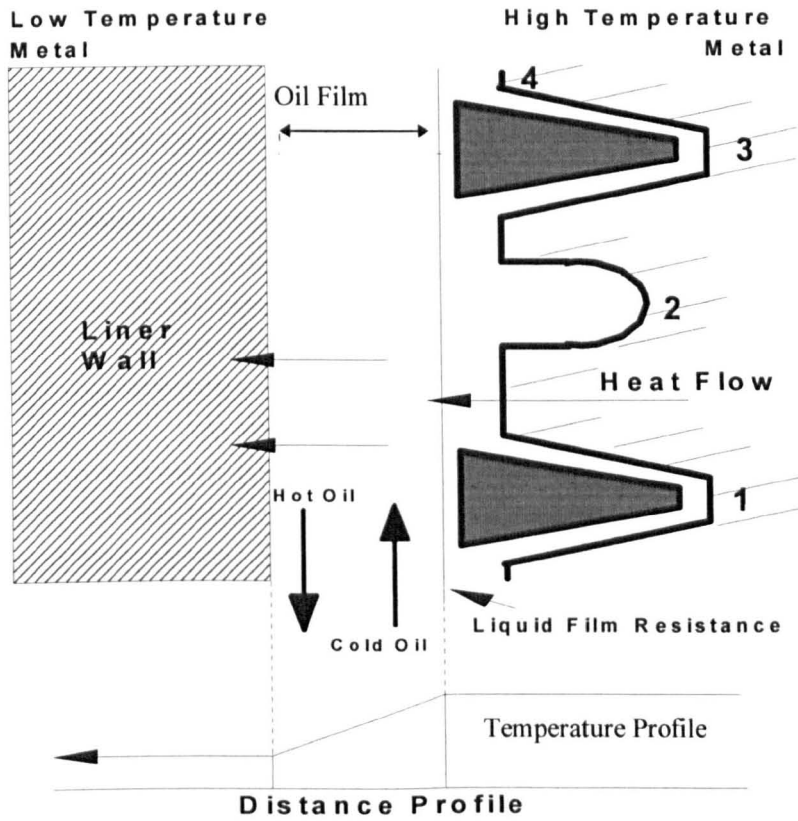

Fig. 2.2 Schematic Diagram of Sampling Positions and Lubricant Flow Regime in the Caterpillar 3406B

As has been stated in section 2.4, the Caterpillar 3406B had four sampling positions. Over the course of the project the sampling system developed from a single point flexible tube system to a multi-point mechanical system with simultaneous sampling capability.

2.5.1 Flexible Tube - Single Sampling Position

The design of the sampling system is shown in Appendix B. The system consisted of 1mm drilled holes at the sampling positions connected with adapters fitted to the under side of the piston crown. A stainless steel micro-bore HPLC tube connected to an adapter, leading down the piston skirt to a clamp, which also secured the continuous PTFE tube to the end of the HPLC tubing. The flexible PTFE tube led around the small end of the connecting rod to a second clamp, with sufficient slack to accommodate radial movement due to rocking of the connecting rod relative to the piston. Great care was taken to ensure that slack/tension in this part of the system allowed sufficient movement without the PTFE tube chafing on the connecting rod small end or the piston crown underside. Two further clamps on the connecting rod led the tube to a bracket fixed to the big end bolt. The tube then exited the engine through the aperture in the engine casing into a sealed box, with a final exit to the sample collecting vessel. The circular motion of the big end created a perturbed sigmoidal motion of the tube, termed 'whiplash', between the big end bracket and the box exit, which was contained within the dimensions of the box. Any erratic lateral movement of the tube within the box was contained by two baffles, between which the tube ran. Initial runs carried out at low speeds, 750-900 rev min^{-1}, proved the concept of sampling but the reliability of the system did not match expectations. Often short running periods could be obtained before the tube failed by fatigue induced shearing at the support bracket. Because the system comprised of one continuous length of PTFE tube, long down-times for repair resulted, with engine dismantling and system replacements.

2.5.2 Modifications to the Flexible Tube - Single Sampling Position

Two modifications eased the sampling system failure problem. One was to split the system into two parts at the support bracket, using a pipe connector. The other was to stiffen the tube at the exit from the bracket connector, thereby spreading the stress concentration. This modification prolonged the running life of the sampling system and enabled the lower portion of the tube to be replaced through the casing aperture.

Running times were significantly extended, albeit at low speeds, but another problem arose in system blockage, eventually traced to the middle and lower clamps on the connecting rod. The clamps were aluminium, whereas the upper one was steel. Dissimilar expansion of the aluminium clamps and the steel connecting rod resulted in the crushing of the PTFE tube after the engine had been running for some time at normal operating temperatures. The clamps were modified and running times were improved further. The sampling system was now proved and viable samples were obtained for analysis. Sampling speeds could now be raised to 1100 rev min^{-1} and the system functioned with minimum interruption on each sampling point in turn.

Above 1100 rev min^{-1}, however, the lower portion of the tube was subjected to increasing inertia forces and the whiplash effect began to tear the tube from the support bracket. Interestingly, the tube itself survived the extreme nature of the whiplash and a reassessment of this part of the run was carried out. The profile of the whiplash showed extreme angles of movement at the fixed ends of the tube with no obvious signs of distress to the tube itself. The flexibility of the PTFE tube easily accommodated the very acute angles imposed. By mounting a swivel in the box close to the engine casing and running the tube through the swivel, the whiplash action was partially relieved. The tube was then able to withstand the forces imposed upon it, and by the ability to move through the swivel, conform more easily to the horizontal element of big end throw.

Running times were now of the order of 8 to 10 hours continuous and at speeds up to 1500 rev min^{-1} without significant failure. Normal operating speeds in commercial use are of the order of 1800 rev min^{-1} and a target for testing had to include this speed. A programme was initiated to achieve this objective, but at 1600 rev min^{-1} the system experienced a complete failure. This was traced to the portion of the system that led over the top of the connecting rod. Despite extreme care in laying the loop between the skirt clamp and the upper connecting rod clamp, the unrestrained portion had stretched under increasing inertia load reversal and destroyed itself against the top of the connecting rod and the piston crown. There was no solution to this problem. The system in its current form had reached the operating limit of its design. To achieve full speed sampling would require a radical redesign, the result of which was the dual sampling system.

2.5.3 The Dual Sampling System

The revised duel sampling system, as the name suggests, took lubricant samples from two positions simultaneously, thereby allowing directly comparable analyses.

The main objective of the redesign was to eliminate, as far as possible, the use of flexible tubing and unrestrained movement of the system. The design concept arose directly from the swivel in the sealing box. In addition, the internal space available and the robust build of the engine gave scope for a purely mechanical system, shown in Appendix C. The thick wall of the piston skirt was sufficient to mount an aluminium suspension unit to a flat surface below the gudgeon pin boss on each side of the piston. The twin forks of the units carried a bushed swivel, drilled and machined to accept the HPLC tube into one end. The HPLC tube was then connected through, and into a carrier pipe, leading to the box swivel. The carrier pipe was itself screwed into the suspension swivel and locked into position.

The end of the carrier pipe was connected to the one piece of flexible tube in the system, which led out of the box into the collecting vessels. The HPLC tube, clamped to the piston skirt, led from each sampling point to each respective suspension swivel, leading through the swivel into a hole drilled into the carrier pipe. The principle of operation was that the suspension swivel would effectively oscillate through a 12° arc around the HPLC pipe. The horizontal and vertical movements of the carrier pipe outlet were restrained by the box swivel and absorbed by the PTFE tube which proved to be able to withstand the stresses.

The major difficulty was obtaining a reliable seal around the HPLC/suspension swivel and the entry of the carrier pipe into the swivel. The solution, Appendix D, was found to be the use of a gland nut and seal. This covered the entry of the HPLC into the swivel. A saddle piece and seal, which covered the carrier pipe connection, were secured by lock nuts. The resulting assembly gave a reliable and consistent performance totalling 650 hours of operation at all engine speeds required by the research.

The gland seal life was in excess of 120 hours, but replacement of the seal could easily be carried out through the engine casing access hole. The seal on the saddle piece was found to

be virtually permanent. Some improvements were made to the system by reducing the size and weight of the suspension units. The advantages of the dual sampling system were:

- directly comparable lubricant analyses from two sampling positions,
- comparable measurement of gas flows and pressures,
- direct comparison and performance of varying ring pack designs,
- extended performance tests of development lubricants due to the reliability and design of the system,
- measurement of lubricant flow within the ring zone, using tracer elements, from two sampling positions simultaneously.

2.5.4 The Bore Wall Sampling System

One further sampling facility was added to the programme, the capability to carry out bore wall lubricant sampling independently of the dual system. The system had access through the engine casing, to the top ring reversal position at Top Dead Centre (TDC), on the inner face of the bore wall. This sampling system was proved over 100 hours of testing but was not used as a regular sampling position.

2.6 Measurement of Oil Mass and Gas Flow in the Caterpillar 3406B

Oil mass and gas flow rates were measured in the ring pack of the Caterpillar 3406B at each sampling position. Initial investigations used pre-1991 US emission regulation ring packs Subsequent work used 1991 and 1994 US emission control ring packs, discussed in Chapter 5.

Using the initial sampling system of the flexible tube with a single sampling position, each sampling position was taken in turn and oil masses and gas flows measured. The engine runs were carried out at three different speeds, 750, 1100 and 1500 rev min^{-1} and lasted for 2 hours. Lubricant was collected from the sampling system by placing the PTFE tube exiting the engine into a glass vial. The lubricant was transported through the PTFE tube as a form of mist/aerosol, and by impinging the PTFE tube onto the wall of the glass vial the lubricant

could condense/coalesce, running down to the bottom of the glass. Oil masses were measured by weighing each collecting vessel before and after each engine run. The total amount of oil collected over the engine run was averaged to give a 'grams / hour' value.

Gas flow rates were more difficult to measure. A pressure gauge was tried initially, but the rapidly fluctuations in gas flow made accurate readings impossible. An alternative was to time the displacement of water from a 1l flask by the sampling gas flow i.e. the higher the gas flow, the shorter the time required to empty the flask. This method was not affected by the fluctuating gas flow and consistent time readings were obtained. These times were then converted into litres/min of gas flow. This measurement was carried out every 15 minutes during the course of the engine run under specified conditions, and the average time taken. The gas flow measurement also monitored the integrity of the sampling system during the course of an engine run. Thus, a steadily reducing gas flow showed the sampling system was blocking and appropriate intervention could be made.

2.6.1 Oil Mass Obtained from Pre-1991 US Emission Control Ring Pack

The initial oil mass flow rate results are shown in Fig. 2.3, varying with sampling position and engine speed and the loads detailed in Table 2.1. The oil mass flow rate generally decreases as the sampling position progresses up the piston, which is as expected.

However unexpected results were also seen. The oil mass flow rate at position 4 was greater than position 3. A possible reason for this was that oil was passing the face of the top ring and not into the piston ring groove, where sampling position 3 was situated. However, the sample obtained from position 4 was not liquid lubricant, but more of an 'oily' soot.

Another interesting point was the effect of engine speed on the oil mass flow rate. As the engine speed (rev min^{-1}) increased the oil mass flow rate decreased. This result was thought to be caused by the following factors;

- firstly, as the engine speed increased the relative load applied by the water brake also increased,
- secondly, the increase in load increased the engine temperature, causing the aluminium piston to further expand in the cylinder bore, thus inhibiting the oil flow into the ring pack and behind the piston rings.
- thirdly, perhaps, more significantly, the increased cylinder gas pressure forced the piston rings closer to the cylinder wall, and also increased gas blow-by behind the rings, forcing oil back down through the piston ring pack and bore, see section 2.6.2.

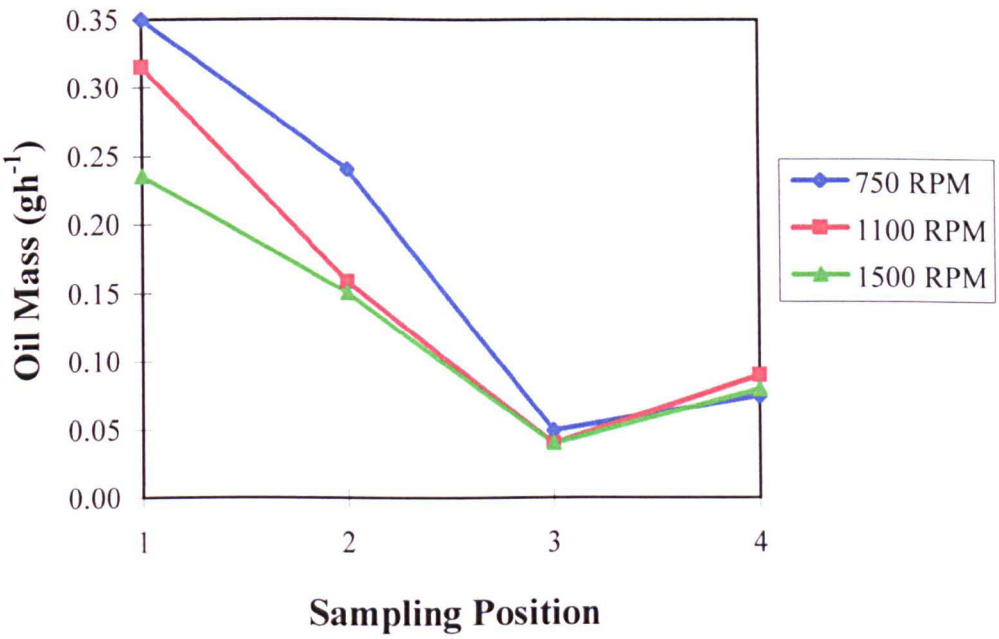

Fig. 2.3 Oil Mass Flow Rate as a Function of Sampling Position and Engine Speed in the Caterpillar 3406B (Load 89 BHP, 95 BHP, 175 BHP)

2.6.2 Gas Flow Rates Obtained from Pre-1991 US Emission Control Ring Pack

The gas flow rates, in relationship to the speed and the loads in Table 2.1, can be seen in Fig. 2.4. From the results it can be seen that as the sampling position progresses up the piston the gas flow rate increases, as is to be expected from the sealing action of the ring pack containing the combustion gases.

Once again, however, anomalies arise. As the engine speed increases the gas flow rate decreases. This also could be due to the increase in load, applied by the water brake, that was associated by increases in engine speed. The reversal in oil mass flow rate between position 3 and 4 was not reflected in the respective gas flows rates, which could possibly arise from the controlling aspect of the ring pack, especially at the top ring.

From examination of these results it was decided that further investigation was needed into the effects of engine speed and load on oil mass and gas flow. This work concentrated on positions 1 and 3, section 2.6.3.

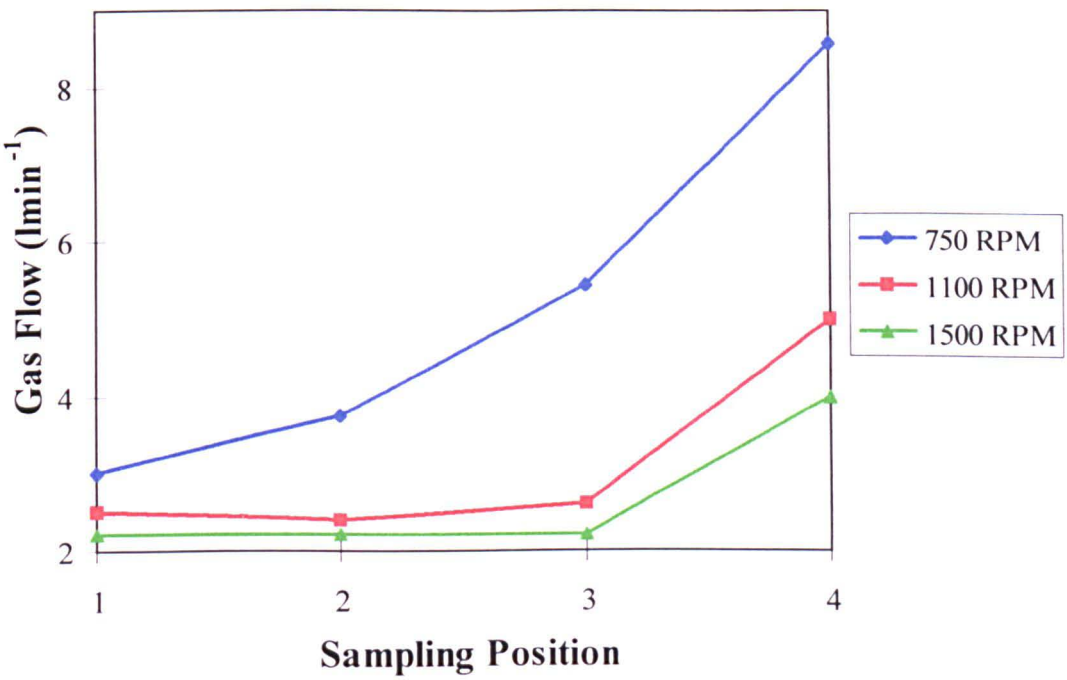

Fig 2.4 Gas Flow Rates as a Function of Sampling Position and Engine Speed in the Caterpillar 3406B (Load 89 BHP, 95 BHP, 175 BHP)

2.6.3 Variations in Oil Mass and Gas Flow Rates as a Function of Engine Speed and Load.

Experiments measured oil mass and gas flow rates in relation to engine speed and load. Using the dual sampling system, oil masses and gas flow rates were measured from sampling positions 1 and 3, for a series of engine runs between 750 and 1500 rev min^{-1}. Three different loads were applied to each engine speed, with the oil masses and gas flow rates measured, as described in section 2.6. It should be noted that both the oil mass and gas flow rates were considered to be indicative, rather than an absolute, due to the design of the sampling system (i.e. internal diameter of the HPLC tubing), which would effect the measured values. Therefore the measured oil mass and gas flow rates should be considered as an indication of trends.

The oil mass flow rate data, Fig 2.5, shows that the rate obtained from position 1 was up to thirty times the rate obtained from position 3. Another point to note from position 1 was that more oil was obtained with increased engine speed, contrary to results seen previously, section 2.6.1. This inconsistency has not been fully explained by this work, but shows that lubricant flow within ring packs is more complex than previously thought. Engine load only appeared to effect the oil mass flow rate at increased engine speeds. At 1500 rev min^{-1} the oil mass increased with increasing load up to 130 BHP, where oil mass flow rate was at its maximum. As the load was increased above 130 BHP the oil mass flow rate decreased. This effect may have been due to the higher speed/load combinations increasing the engine temperature, causing the piston to expand further into the bore, thus restricting oil flow.

The data obtained from position 3 proves harder to interpret. The oil mass flow rate was initially high at 750 rev min^{-1}, decreasing to a minimum at 1100 rev min^{-1}, increasing to a maximum at 1300 rev min^{-1} and then reducing again at 1500 rev min^{-1}. Contradictory to position 1, load seemed to have little effect on the oil mass flow rate obtained from position 3. The graph is more difficult to interpret due to the small oil masses involved, however there are consistent 'peaks', which appear at 900 rev min^{-1} (85 BHP), 1100rev min^{-1} (100 BHP), 1300rev min^{-1} (130 BHP) and 1500rev min^{-1} (160 BHP). These 'peaks' show a potential trend in the oil mass, which could be a function of engine speed and load and/or a

function of the piston ring pack performance. It should also be noted that position 1 also shows similar 'peaks', albeit to a lesser extent.

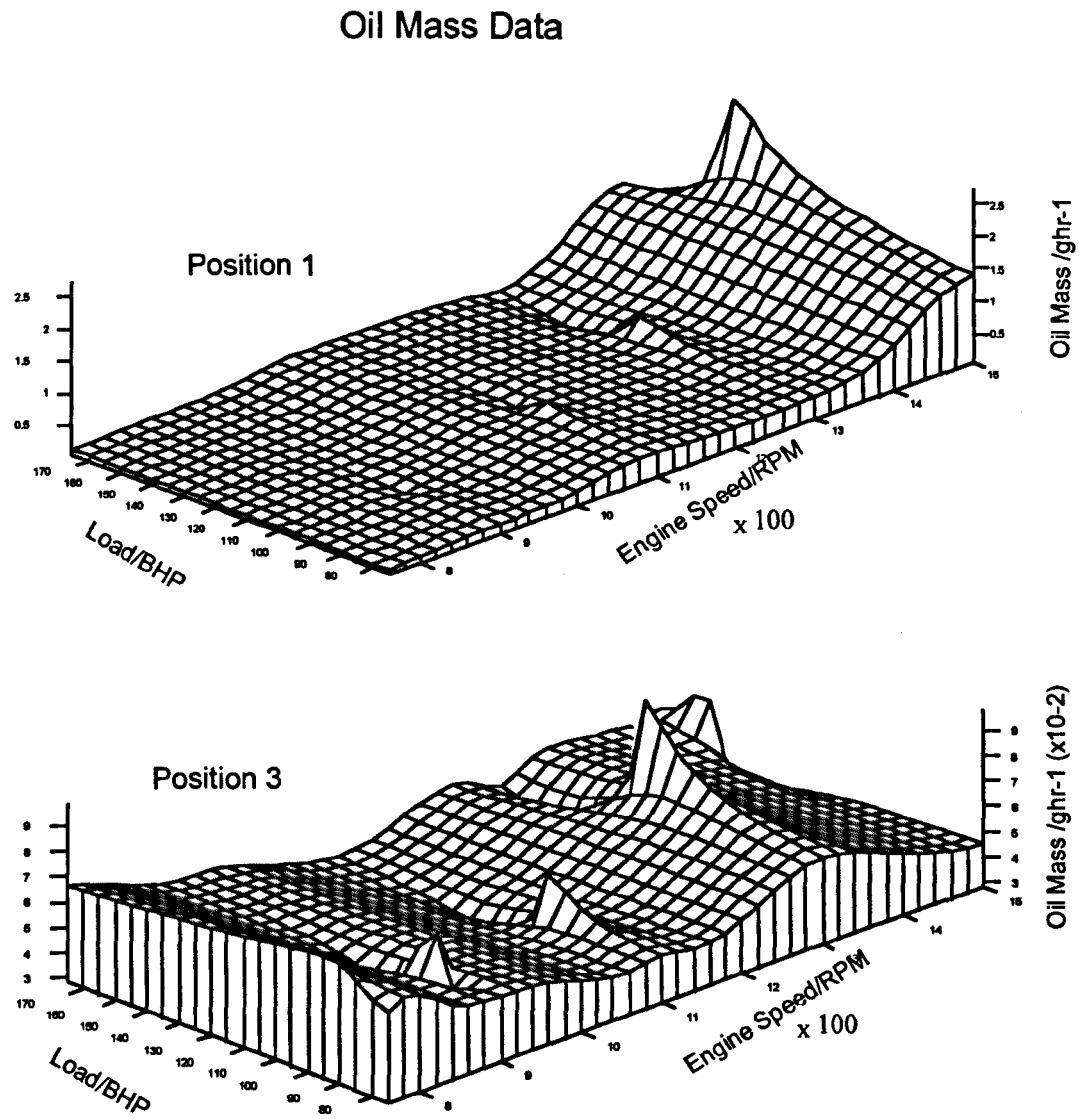

Fig. 2.5 Variation on Oil Mass Flow Rate from Positions 1 and 3 as a Function of Engine Speed and Load Obtained from the Caterpillar 3406B

Gas flow rate data is shown in Fig. 2.6. The measured gas flows rates were greater for position 3, up to three times the measured flow from position 1. The data obtained from position 1 showed that the gas flow was at its lowest at 750 rev min^{-1}, increasing with engine speed and reaching a plateau between 1100 rev min^{-1} and 1500 rev min^{-1}. Engine load did not appear to affect the gas flow. The data obtained from position 3 showed very

similar trends. The increase in gas flow to a plateau shows the increased combustion pressure associated with increasing engine speed and also the sealing action of the piston ring packs. It should be noted that there are two 'peaks' at 1100 rev min^{-1} (90 BHP) and 1500 rev min^{-1} (140 BHP), which correspond to 'troughs' in the obtained oil mass, Fig. 2.5. It can also be noted that the 'peaks' in oil mass, Fig. 2.5, correspond to 'troughs' in the measured gas flow. This phenomenon highlights the complex relationship between measured oil mass and gas flow in a modern diesel engine.

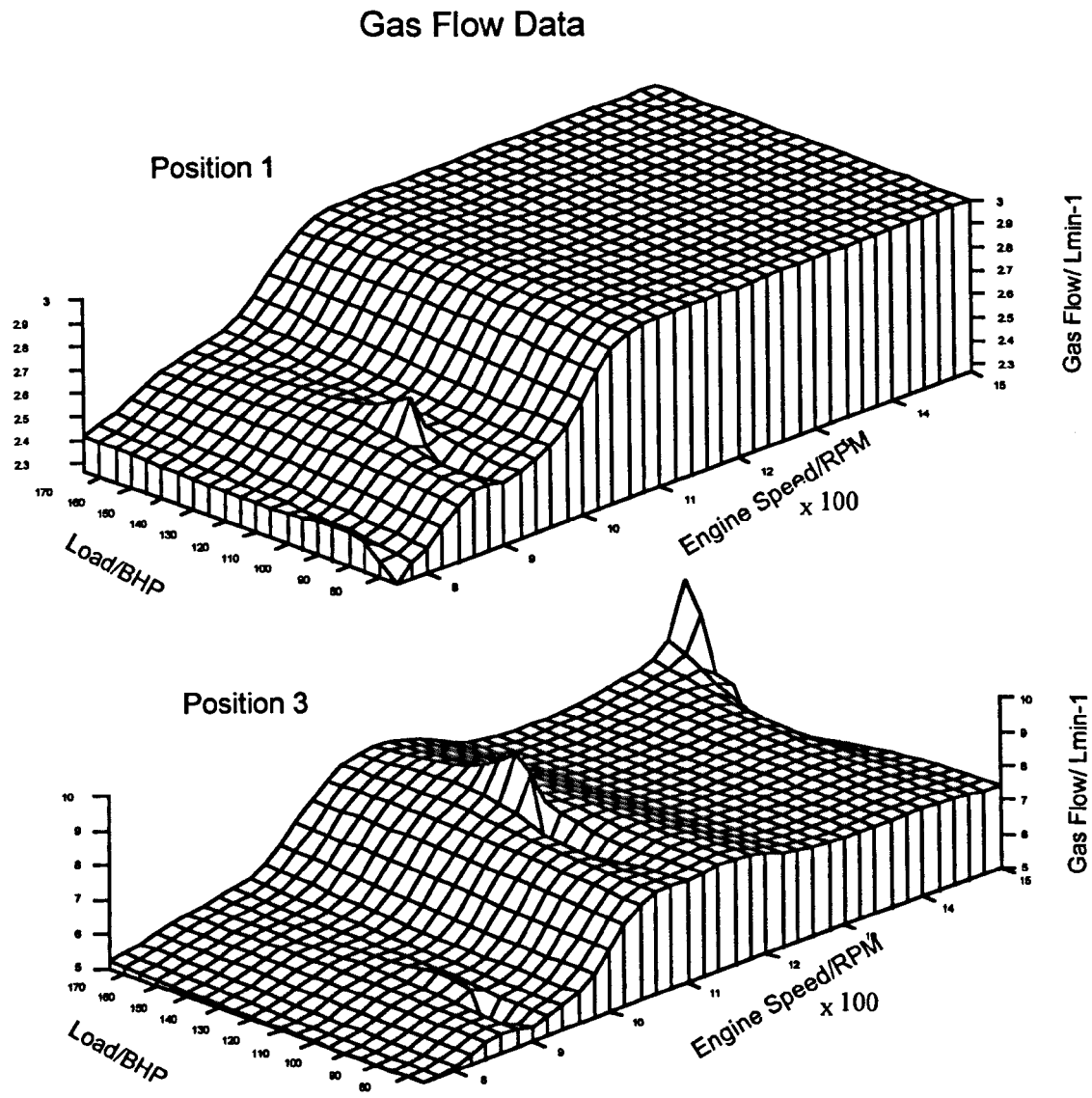

Fig. 2.6 Variations in Gas Flow Rate as a Function of Engine Speed and Load Obtained from the Caterpillar 3406B

2.6.4 Variation in Oil Mass Flow Rate with Time

An equilibrium system is implicitly assumed to model ring pack oil flow[2.8, 2.9]. However, long term sampling for oil mass and gas flow rates showed a non-equilibrium system, with a symmetrically opposite trend, Fig. 2.7. This behaviour was reproducible for sampling position 1, but far less noticeable for the other sampling positions. The cause of this phenomenon is not totally clear, but may be associated with varying rates of piston ring rotation.

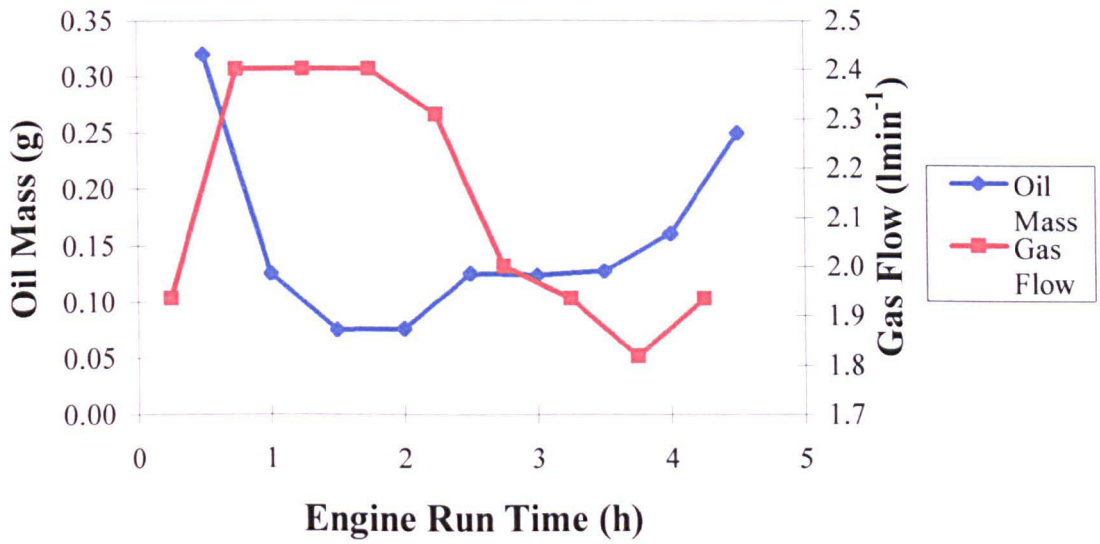

Fig. 2.7 Variations in Oil Mass and Gas Flow with Engine Run Time from Sample Position 1 Obtained from the Caterpillar 3406B, at 1200rev min^{-1}, Load 104BHP

Fig. 2.7 shows that as the oil mass flow rate **decreases** the gas flow rate **increases**, which could possibly be attributed to the action of oil flow through the sampling system (note: the sampling system consisted of a tube of internal diameter 2 mm). It was found, particularly with position 1, that oil sometimes travelled down the PTFE tube in 'plugs'. This fact was attributed to the ring gap on the 2nd ring, lining up with the sampling hole at position 1. It was found that the oil 'plugs' inhibited the gas flow rate, hence showing a reduced gas flow rate with the experiments. However, when the oil travelled down the tube in a mist, the gas flow rate was less inhibited, thus showing an increased gas flow rate. This effect emphasised that all subsequent oil mass measurements must be carried out under equilibrium conditions, i.e. when the oil flow was in the form of a mist/aerosol.

2.7 Oil Control Ring

As piston ring pack design becomes more stringent in controlling oil flow, the integrity of the assembly and installation of the ring pack becomes ever more important. The oil scraper ring is reliant on fine-edged rings, which must not be damaged when installing the piston into the bore.

Typical oil mass flow rates obtained from the pre-1991 US emission control ring packs were in the region of 0.5 gh^{-1}, for position 1, to 0.03 gh^{-1}, for position 4. One engine test run produced oil masses twenty times greater than was previously seen, which was thought suspect. The engine was dismantled and the piston removed for investigation. Close investigation of the oil control ring found a very small 'blemish', very difficult to see by eye, but which could be felt by running a finger across the ring. The oil control ring in the Caterpillar 3406B is a double spacing 'razors edge' design which greatly inhibits the flow of lubricant into the piston ring zone. However, a very small 'blemish' allowed significantly greater quantities of lubricant into the piston ring zone, which was subsequently sampled.

Further to this point, discussions with the engine component assembly industry showed that individual engines, with high oil consumption, can often be related via quality assurance/control (BS 5750/ISO 9000) to relatively inexperienced assembly workers. Due to their relative inexperience in the job they were consistently damaging oil control and piston rings when fitting pistons into cylinder bores.

The damage incident to the oil control ring highlighted the performance of the sampling system. At first the low oil mass flow rates obtained from the Caterpillar 3406B were attributed to problems associated with the sampling system, and that this was the rate determining step. However, due to the damaged oil control ring, it was found that the rate determining step was the small amount of lubricant in the piston ring zone when the oil control ring was undamaged. Once a new oil control ring was fitted, oil masses were of the reduced order seen previously.

This observation leads to the conclusions that:

- extreme care has to be taken when fitting the oil control ring, so that no damage occurs, if good oil control is to be maintained and that subsequent oil related exhaust emissions are to be kept low,
- the sampling system would take a representative portion of whatever amount of lubricant was present in the piston ring pack.

2.8 A Physical Representation of Lubricant Volume in the Piston Ring Pack of the Caterpillar 3406B Test Engine

The nature of the research project has meant that the cylinder head on the Caterpillar 3406B has been removed frequently. This has shown interesting results in terms of the amount of lubricant present in the piston ring pack. After the cylinder head was removed it was generally found that the cylinder bore was almost 'dry' of lubricant, with very little oil present on the face of the piston rings or behind the piston ring grooves. It was noted that the top ring was particularly 'dry' of lubricant. This observation has shown that 'low emissions' engines, such as the Caterpillar 3406B, have very little lubricant in the piston ring zone, which leads to the conclusion that the lubrication regime in the piston ring pack, especially the top ring, must either be thin film lubrication or more likely, boundary lubrication.

During the course of this work liquid lubricant in any substantial amount has only been found on the face of the top ring twice. Firstly, when the oil control ring was damaged which lead to a substantial increase in oil volume allowed into the piston ring zone, as described in section 2.7. Secondly, when a lubricant of very low volatility, MRI-1[2.9], was ran through the engine. The MRI-1 lubricant will be discussed further in section 2.18.

2.9 Temperature Profile of the Caterpillar 3406B Piston

Measurement of the metal temperature profile of the sampling piston was carried out using Templugs[R], purchased from VernoLab, France. Templugs[R] are screw plugs, which harden in

relation to both the temperature they were subjected to and also the length of time of exposure. Therefore the hardness indicates the highest temperature the Templugs[R] were subjected to. For these temperature measurements the sampling piston was modified to have eight holes drilled and tapped from sampling position 4 to the bottom of the piston skirt, so as to accommodate the plugs. The Templugs[R] were fitted into the piston, making sure they were flush with the piston face. For these experiments the engine was operated at 1500 rev min^{-1} at 170 BHP, for 4 hours under normal operating temperatures. After the engine run, the engine was dismantled and the Templugs[R] removed and sent to VernoLab for analysis. The plug's hardness were analysed and related to the temperature they had been subjected to. The temperature profile of the sampling piston can be seen in Fig. 2.8.

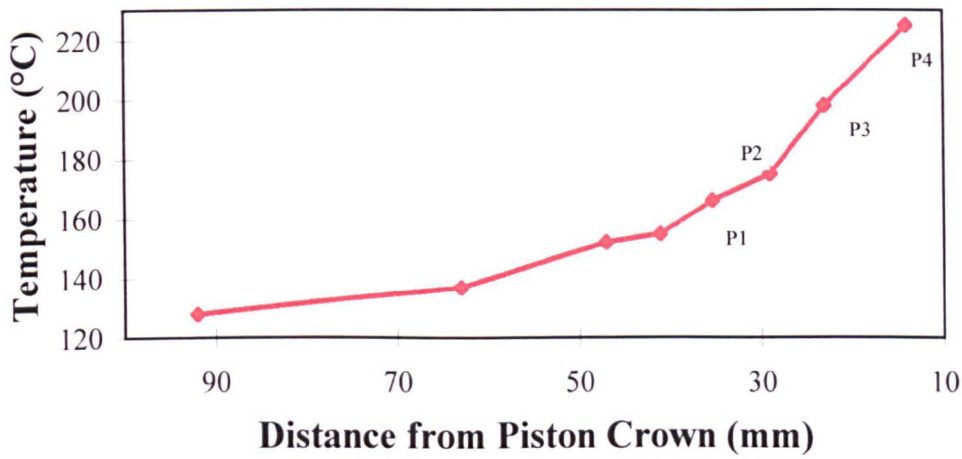

Fig. 2.8 Temperature Profile of the Caterpillar 3406B Sampling Piston (Engine Speed 1500 rev min^{-1}, Load 170 BHP)

The piston temperature reduces as the Templugs[R] position moves further away from the piston crown. Important points to note are the temperatures relating to the sampling positions, Table 2.3. These temperatures, relating to lubricant volatility will be discussed in section 2.15.5.

Sampling Position	Temperature / °C
1	166
2	175
3	198
4	225

Table 2.3 Caterpillar 3406B Piston Temperature Compared to Sampling Position (Engine Speed 1500 rev min^{-1}, Load 170 BHP)

2.10 Pinned Piston Rings

Section 2.6.4 highlighted that piston rings can rotate in their grooves. As an experiment, the piston rings were pinned in place to see if this affected the amount of lubricant obtained from the sampling system. The second ring and top ring on the Caterpillar 3406B were 'pinned' to inhibit the rings rotation. The pinned ring samples gave considerably less lubricant, Table 2.4. The resulting samples also showed increased chemical degradation, which was attributed to less sample being obtained from the sampling positions.

Sampling Position	Rotating Rings Average Mass of Oil (gh^{-1})	Pinned Rings Average Mass of Oil (gh^{-1})
1	0.44	0.21
3	0.09	0.01

Table 2.4 Average Hourly Oil Mass Comparing Pinned and Rotating Piston Rings from the Caterpillar 3406B, at an Engine Speed of 1200 rev min^{-1} and a load of 104 BHP

It can be clearly seen that pinned piston rings allowed less oil to be present in the piston ring grooves at the sampling points. Upon engine dismantling, carbon deposits were found in the top piston ring grooves and on the back face of the top ring. Such deposits were not present when the rings were allowed to rotate. These carbon deposits suggest that either considerable chemical breakdown of the lubricant has occurred, or that there had been a build up of solid combustion products or a combination of both. These deposits had not been removed during engine operation because the piston rings were not free to rotate.

2.11 Measurement of Oil Transport from the Sump to Sampling Positions

Experiments were carried out to measure lubricant transfer times from the sump to the four sampling positions. To measure lubricant transport, a tracer element was quickly added to the sump, so that lubricant transport could be monitored through the ring pack.

Previous workers[2.10] had used either a radioactive element (^{32}P in tributyl phosphate) or a magnesium based detergent to measure lubricant transport from the sump to the top ring for two diesel engines, a Petter AA-1 and a Caterpillar 1Y-540. Results showed the transport time to be 3 minutes for the Petter AA-1 and 3-5 minutes for the Caterpillar 1Y-540.

2.11.1 Choice of Tracer Element

Because of lubricant leakage, the use of a radioactive tracer element was deemed unsuitable on Health and Safety grounds, so a magnesium based detergent was considered. A suitable commercial SAE 15W-40 lubricant, with a calcium based detergent, was chosen for the test. A concentrated magnesium based detergent (magnesium sulphonate) could, therefore, be used as the tracer element.

2.11.2 Theory of Technique

The operating concept for the tracer technique was to establish equilibrium engine running conditions and then inject the tracer element into the sump. Measurement of the tracer element would require frequent sample collection from the four sampling positions. The lubricant obtained could then be analysed by suitable techniques, such as Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), for magnesium concentration. Previous results had shown that shortly after injection the tracer was seen in the lubricant sample, rising to a maximum and maintaining its level, showing a typical sigmoidal curve.

Certain assumptions were made to undertake the tracer experiments;

- that instantaneous mixing of the tracer with the sump oil would occur,
- that 1mg of oil would be collected in the sample vessel for each sample.

2.11.3 Calculation to Determine Magnesium Concentration in the Sump

It was necessary to determine the amount of magnesium sulphonate additive to be added to the sump oil so as to give a measurable concentration of magnesium in the ring zone samples obtained. After consideration, it was decided that the final magnesium concentration in the sample should be of the order of 1ppm (parts per million by volume), to give reasonable detection limits for the ICP-OES technique. To achieve this concentration, certain points needed to be considered;

- to undertake the analysis, the oil sample must be diluted with 2ml of a suitable solvent (i.e. ICP grade white spirit). It was, therefore, this sample that needed a magnesium concentration of 1ppm. The sump contents would, therefore, be much higher in magnesium concentration.
- assuming that 1mg of oil would be obtained was not accurate enough, actual sample masses would need to be measured. Differences in sample mass could then be used to correct the magnesium concentration.

Calculation of the magnesium concentration in the sump, which would lead to a 1ppm magnesium concentration in the diluted sample, was:

The average oil mass obtained per minute for each sample: $1mg = 1\times10^{-3}g_{oil}$

The amount of oil in vessel after 2ml dilution step $= 1\times10^{-3}g_{oil} \div 2ml_{dilution}$

$$= 5\times10^{-4}g_{oil}ml^{-1} \cong 500ppm_{oil}$$

The sump capacity of the Caterpillar 3406B = 31.5L = 31500ml

The amount of this oil needed to be a tracer, therefore = 31500ml × $500ppm_{oil}$= $63ml_{oil}$

However the magnesium content in the magnesium sulphonate additive is 10%, so the figure needed to be corrected to give 100% magnesium:

$$\therefore 63g_{oil} \times (100 \div 10) = 630g_{oil}$$

Using assumption: $1g_{oil} \cong 1ml_{oil}$ (Actual figure $0.84g_{oil} = 1ml_{oil}$)

$$\therefore 630g_{oil} \cong 630ml_{oil}$$

Therefore, 630ml of magnesium sulphonate additive needed to be added to sump, to give 1ppm magnesium in sample vessel after dilution.

2.11.4 Injection of Marker Techniques

The next consideration was to effectively inject the magnesium sulphonate additive into the sump of the engine. Four possible injection techniques were considered for their advantages and disadvantages.

Technique 1: To inject the oil/additive mix through the oil filler cap situated on the top of the engine. However, the mixture would not instantly reach the sump. Also, there was uncertainty that all of the mixture would reach the sump at the same instant.

Technique 2: To remove the contents of the sump, placing them into an oil barrel and then adding the oil/additive mixture. The barrel contents would then be vigorously shaken and placed back into the engine via the oil filler cap. The method would ensure good mixing of the additive with the sump oil. However, this method would entail turning off the engine, draining the sump of oil, mixing the sump oil with the additive and then placing the contents back into the engine. This would mean that the engine would not be running under equilibrium conditions when the mixture was added. Also, from engine start-up more oil

could be seen in the piston ring pack due to the engine not being at its normal operating temperature, and therefore it may possibly give false results.

Technique 3: To inject the oil/additive mixture through the oil drainage pipe at the bottom of the sump. This method would require a high pressure pump to push against the flow of oil coming out of the sump. This injection technique could also take a long time to carry out.

Technique 4: To inject the oil/additive mixture down the oil dipstick tube, which would place the additive into the bottom of the sump, immediately adjacent to the oil pump intake. Injecting the mixture into the bottom of the sump seemed the most favourable method, because it would be dispersed around the sump very quickly. Injection would also require little effort.

For the first experiment **technique 4** was chosen, because it seemed the quickest and simplest injection technique. It was found that the viscous nature of the additive would make direct injection difficult, so mixing of the additive with the lubricant under test was carried out. The experimental apparatus consisted of a 3L glass beaker, a 3L plastic beaker, a hot plate, a stirrup pump and a plastic tube which would connect the stirrup pump to the dipstick tube.

2.11.5 Experimental Procedure (Engine Run)

1. The sample collecting vessels were labelled and weighted before testing began.
2. The engine was ran for 1 hour at 1500 rev min^{-1} to allow equilibrium conditions to be achieved.
3. 630ml of magnesium sulphonate additive was placed into a 3L glass beaker. 1370ml of sump oil was added to the additive. The beaker was placed onto a hot plate and heated to 80-90°C (approximate sump temperature), whilst being thoroughly mixed.
4. With the engine running samples were taken for 6 minutes, to give a 'base line' before the oil/additive mixture was injected. After 6 minutes the oil/additive mixture was injected, using the stirrup pump, down the dipstick tube, in less than 60 seconds.

5. Samples were taken every 1 minute for position 1 and every 3 minutes for position 3. Samples were collected for 60 minutes.
6. The sample vessels were weighed, so the mass of oil in the vessel could be found.

2.11.6 Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

ICP-OES is a well established analytical technique, using ionised argon gas to produce a plasma. The plasma is sustained in a three tube quartz torch, placed in a electro-magnetic field around a copper coil. The copper coil is connected to a frequency generator. Samples enter the machine via tubing attached to a peristaltic pump, which pulls the samples into the machine, and into the nebuliser. An aerosol from the sample is generated by the nebuliser system and is carried by a small flow of argon into the plasma where the elements in the sample are atomised and excited. These excited atoms decay to lower energy states, releasing quantum's of energy. The resulting emissions from the elements are directed into a spectrometer where optical dispersion of the emitted light and detection takes place. The detection of a specific element is possible due to each element having a specific wavelength in the electromagnetic spectrum where it releases energy (i.e. magnesium is at 238.204nm). The concentration of the element being measured is directly proportional to the emitted energy. ICP-OES can be used to measure multiple elements to very low detection limits, within a short space of time. Detection limits are dependent on the element under test, but are typically in the parts per billion (ppb by volume) range.

2.11.7 Experimental Procedure (ICP-OES)

The equipment used was a Perkin Elmer 'Plasma 40' ICP-OES. The equipment was set up and calibrated to measure magnesium concentrations. The solvent used to dilute the samples was ICP grade white spirit.

The testing procedure carried out is shown below:

1. The machine was switched on and the computer software set to measure magnesium at a wavelength of 238.204nm.

2. The plasma torch was ignited to produce the ionised argon gas plasma and left to stabilise for 45 minutes.
3. The machine was calibrated by testing a blank solution of white spirit, and a standard solution of 1ppm magnesium made up in white spirit.
4. Each sample was taken in turn and diluted with 2ml of white spirit, aspirated and then run. The analysis for each sample took approximately 60 seconds. Results were printed out via the printer attached to the machine.
5. The machine was re-calibrated after every ten samples by repeating step 3.
6. The results obtained, in ppm, were the raw magnesium concentration.

2.11.8 Data Presentation

The raw magnesium concentration data was placed into a database, which already contained the oil mass data. The magnesium concentration was then corrected for oil mass using a simple ratio relationship. The data was then subjected to a smoothing correction to smooth the data and also to take into account that each sample had an effect on each other (i.e. the time to change sample tubes took up to 20 seconds). The corrected data was then plotted on a graph of corrected magnesium concentration versus time, Fig. 2.9.

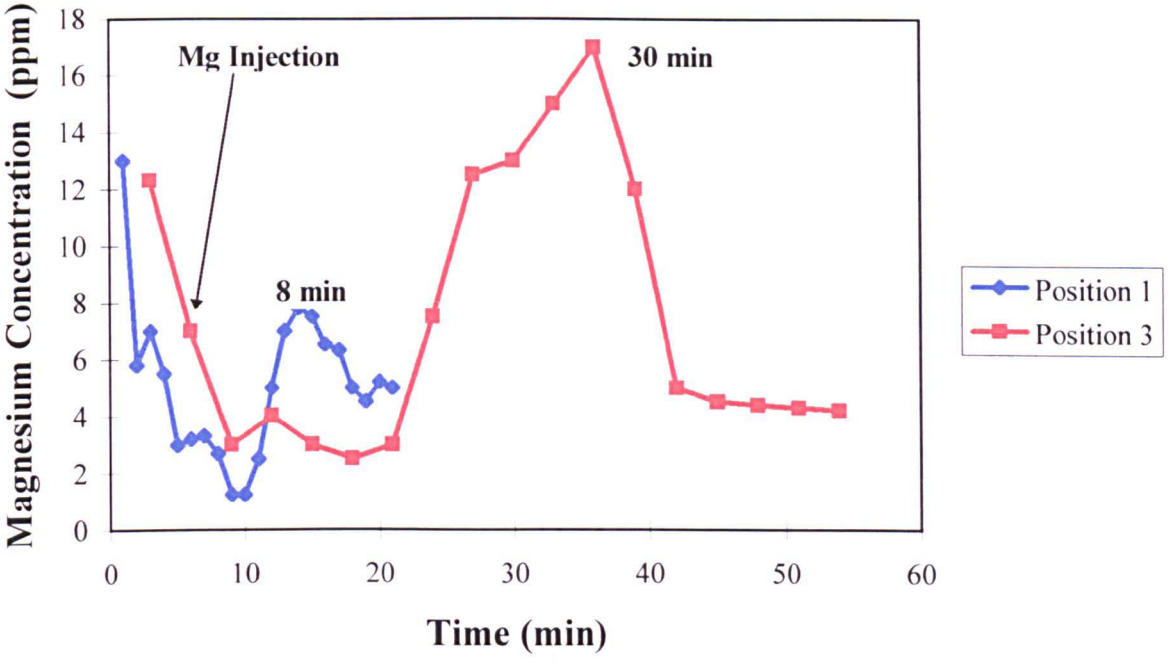

Fig. 2.9 Comparison of Magnesium Transfer from the Sump to Sampling Positions 1 & 3 (Caterpillar 3406B - 1500 rev min^{-1}, Load 130 BHP)

The results show that the oil/additive mixture after injection took 8 minutes to reach a maximum for position 1 and 30 minutes to reach a maximum for position 3. Previous results[2.11] for engines that did not have environmental considerations in their ring pack design produced results between 3 and 5 minutes to reach the top ring (position 3). The time for the lubricant to reach the top ring of the Caterpillar 3406B, therefore, shows almost a tenfold time increase. This highlights that lubricant flow to the ring pack in 'low emissions' engines is far slower than previous engines which are not classed as 'low emission'.

The shape of the produced graph was very unexpected. The magnesium concentration did not show a sigmoidal relationship, as had been seen previously, with the magnesium concentration reaching a maximum and then tailing off. The cause of this was first not apparent, but was thought to be caused by the manner of the 'marker' addition.

The mixture, injected down the dipstick, appeared to have not been dispersed as had been implicitly assumed. Upon investigation it was found that the bottom of the dipstick tube was

just below the oil pump intake. It was therefore thought that the mixture was not being allowed to disperse in the sump, because it was immediately being transferred into the oil pump. Therefore, the magnesium additive was remaining as a concentrated 'plug'. It could be seen that this magnesium 'plug' was rising to a maximum and then tailing off as the plug travelled down from the piston into the sump. 'Plug flow' in a circulating system which is not strongly mixed is a well known phenomenon in process technology. Further work carried out using magnesium tracer techniques will be discussed in Chapters 3 and 5.

2.12 Test Lubricants

A sampling matrix was organised to investigate how oil formulations of differing quality would perform using the variables of sampling position, engine speed, engine load and diesel fuel sulphur content. Fourteen lubricant formulations were tested in the Caterpillar 3406B under various conditions, Table 2.5.

Lubricant Code	Viscosity Grade	Comments	Engine Speeds Ran / rev min^{-1}	Sampling Positions Ran	Fuel Sulphur Content / %
CAT Oil	SAE 15W/40	API CF-4, CE Caterpillar Recommended	1200	1,2,3,4	1
A	SAE 10W/30	Mineral Based	750, 1200, 1800	1,2	1
B	SAE 10W/30	Mineral Based	750, 1200, 1800	1,2	1
C	SAE 10W/30	Modified Mineral Based	750, 1200, 1800	1,2,3	1
D	SAE 10W/30	Synthetic - No VII	750, 1200, 1800	1,2,3	1
E	SAE 10W/30	Synthetic - With VII	750, 1200, 1800	1,2,3	1
F	SAE 15W/40	Lubricant used for Tracer Experiments	1500	1,2,3,4	1
G	SAE 10W/40	Mineral	1300	1,3	0.3, 0.05
H	SAE 10W/40	Semi-Synthetic	1300	1,3	0.3, 0.05
I	SAE 10W/40	Synthetic	1300	1,3	0.3, 0.05
J	SAE 15W/40	Mineral	1300	1,3	0.18, 0.042, 0.009
K	SAE 15W/40	Semi-Synthetic	1300	1,3	0.18, 0.042, 0.009
L	SAE 15W/40	Synthetic	1300	1,3	0.18, 0.042, 0.009
MRI-1	SAE 30	Synthetic - Very Low Volatility	1800	1,2,3,4	1

Table 2.5 Test Lubricants used for Research[1].

[1] A-E have similar additive packs. F is a commercial lubricant. G-I have similar additive packs. J-L have similar additive packs.

2.13 Infra Red Analysis

Infra red (IR) analysis can identify molecular structures and functional groups if a change in their dipole moments, caused by molecular vibrations, occurs. An IR spectrum is a plot of radiation intensity absorption against frequency, being specific to the molecules in the sample. The IR spectrum produces a 'fingerprint' of the chemical compound in the sample.

Infra red analyses are a well recognised[2.12] test for characterising new and used lubricants. It can also be used to monitor additive depletion, lubricant degradation products and contaminate build up. Fourier Transform Infra Red (FT-IR) has become a powerful tool in the analysis of lubricants. Using the Coates and Setti[2.13, 2.14, 2.15] difference spectra approach, differences between new and used lubricants can easily be quantified. Using computer-based software 'peaks' and 'troughs' in the difference spectra can be integrated to give absorbance values. Therefore, the increase in contaminants or decrease in additive concentrations can be monitored. Research[2.16, 2.17] has used FT-IR as an important condition monitoring tool to monitor lubricant condition and to provide advanced warning of impending problems. Studies have also claimed to use FT-IR to measure physical properties, such as base number[2.18].

2.13.1 FT-IR Experimental Procedure

Sample analyses were carried out between 4000-400 cm^{-1} on a Nicolet 5DXR FT-IR spectrophotometer using a potassium bromide (KBr) 0.1mm path length cell. The KBr cell is ideal for this scanning range because it does not itself show strong absorption's. The FT-IR analyses were carried out in the following manner.

1. The KBr cell was cleaned with cyclohexane.
2. A background spectra of the KBr cell was recorded, to be automatically subtracted from any subsequent analysis.
3. A lubricant sample was pipetted into the cell and pulled through the cell by attaching a vacuum. Approximately 100mg of lubricant was needed to fill the cell. The oil film was checked for the absence of air bubbles.

4. The cell was placed into the infra red beam and the absorption spectra recorded and stored in a computer file.
5. The cell was cleaned with cyclohexane and steps 3-4 repeated.
6. Difference spectra were obtained by subtracting a fresh oil spectra from a used oil spectra and then plotted.

Using the Nicolet computer software, the 'peaks' and 'troughs' of the spectra could be integrated to give an absorbance reading. Table 2.5 shows typical regions in the IR spectra of importance to lubricant condition monitoring.

Infra Red Region cm^{-1}	Component	Nature of Vibration
3700-3000	Water	O-H
1800-1650	Carbonyl Oxidation	C=O
1646-1622	Nitrate Esters	$RONO_2$
1530, 1330-1270	Carbonate	CO_3
1200-1050	Oxidation, Sulphates	C-O-C, SO_4
1030-940	ZDDP (Anti-wear)	P-O-C
850	Fuel Dilution (Gasoline)	Aromatic Hydrocarbons
690-650	ZDDP (Anti-oxidant)	P=S

Table 2.5 Spectroscopic Locations of Lubricating Oils, their Additives and Contaminants

2.13.1 FT-IR Integral Results for CAT Oil

One of the prime objectives of lubricant formulation is to protect the base oil from oxidation during service, achieved by using various additives, such as zinc dialkydithiophosphate (ZDDP). Like many other lubricant additives, ZDDP performs more than one function. Two important functions are its anti-oxidant and anti-wear properties. These two functions are mainly performed by separate fragments of the ZDDP molecule. The depletion of P=S can be related to the ZDDP molecule acting as an anti-oxidant, whilst the depletion of P-O-C can be related to the ZDDP molecule acting as an anti-wear additive. Fig. 2.10 compares oxidative degradation of the base oil with depletion of the ZDDP additive.

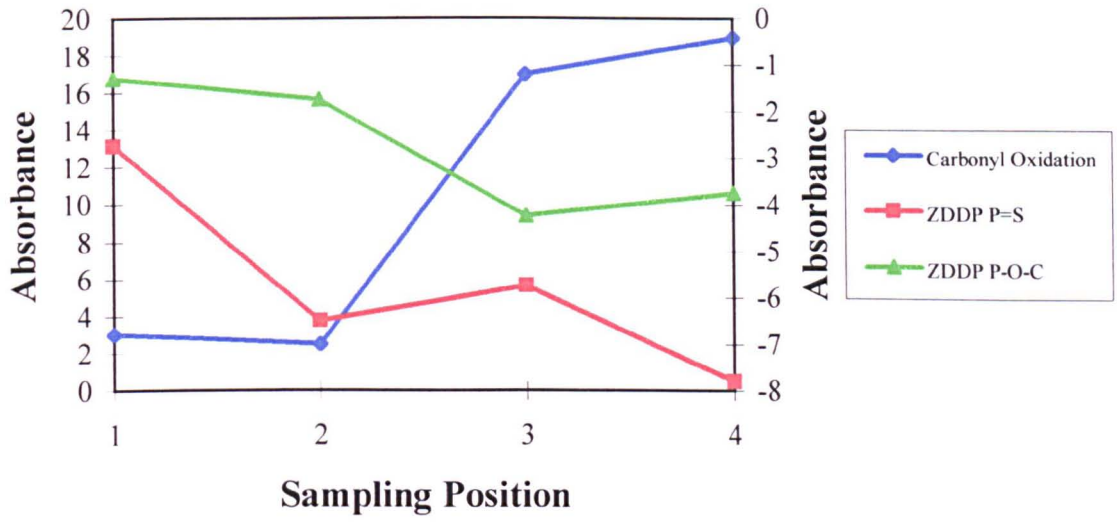

Fig. 2.10 Comparison of Measured FT-IR Integral Intensities (CAT Oil) from Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

Degradation of the P=S part of the ZDDP molecule was moderate at position 1, increasing sharply between position 1 and position 2. Degradation was less pronounced between positions 2-4. In contrast the carbonyl oxidation, used as a measure of base oil oxidation, did not increase sharply until position 3-4. These results appear to show that the P=S depletion protected the base oil from oxidising at positions 1 and 2, but was either inactivated or exhausted at positions 3 and 4, where substantial oxidation was measured. The measurement method of sampling and analysis shows promise as a comparative study of ZDDP activity, as a function of base oil oxidation.

Depletion of the P-O-C part of the ZDDP molecule follows a different trend. Significant depletion only occurred at position 3 and 4, with very little difference between positions 1 and 2. ZDDP P-O-C is the anti-wear part of the ZDDP molecule. High localised temperatures cause the ZDDP compound to split into fragments (i.e. ZDDP P-O-C). This could explain why more P-O-C was depleted at positions 3 and 4, where the temperatures were higher, than positions 1 and 2 (ref: Section 2.9).

Nitrate ester formation and carbonate depletion, the latter from overbased detergent additives, show complementary trends, Fig. 2.11. Nitrate esters are formed by the reaction of combustion generated nitrogen oxides reacting with the base oil. Carbonate depletion is due to the reaction of the basic detergent additive with the acidic nitrogen and sulphur oxides in the combustion gases. Considering successive sampling positions up the piston, there is an increase in nitrogen and sulphur oxide concentrations, which leads to an increase in nitrate ester formation and carbonate depletion.

C-O-C oxidation and sulphate formation absorptions were measured together, having proved difficult to separate. The reason was that they were both present in large concentrations and being adjacent in the IR region they overlap strongly. However, clear distinctions were shown between sampling positions 2 and 4 (higher oxidation/sulphate formation) on the piston face and positions 1 and 3 (lower oxidation/sulphate formation) behind the second and top piston ring. This trend was repeated in other analysis techniques such as kinematic viscosity, section 2.14, and thermal analysis, section 2.15.

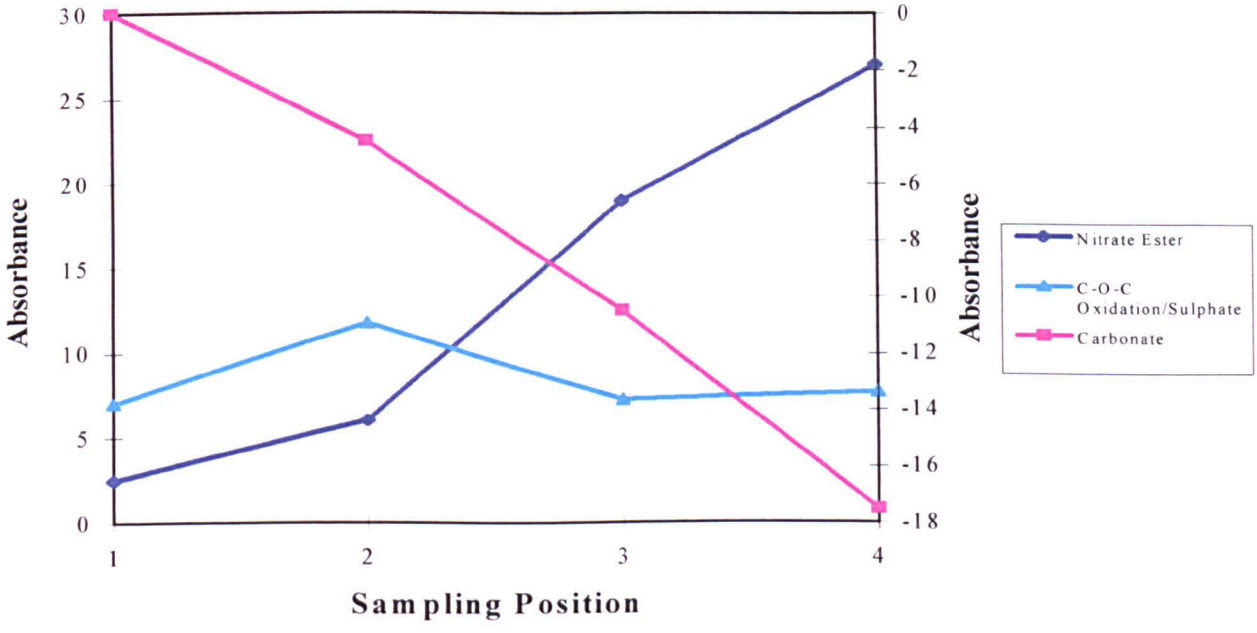

Fig 2.11 Comparison of Measured FT-IR Integral Intensities (CAT Oil) from Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

2.14 Viscosity Measurements

Viscosity is defined as resistance to flow, where this resistance is dependent on the intermolecular forces within the liquid. These forces determine the mechanical resistance when one layer of liquid slides over one another, thus providing resistance when one molecule moves past another. Two types of viscosity measurement are readily available for lubricant viscosity measurement:

- Dynamic or absolute Viscosity. This is usually measured with rotary viscometers in which a rotor spins in a container of the fluid and the resistance to rotation (torque) is measured.
- Kinematic Viscosity. This measures the rate of flow of a liquid through a capillary tube under the influence of the constant force of gravity. The dynamic viscosity can be calculated by multiplying the kinematic viscosity by the density of the lubricant.

2.14.1 Kinematic Viscosity Measurements

Kinematic viscosity measurements were carried out using the Stanhope-Seta SetaVis microviscometer. The machine was ideal for measuring ring zone samples due to the small amount of sample needed to carry out a test, no more than 0.3ml. The SetaVis had a heated bath, filled with a low viscosity fluid (Seta technical white oil). Six viscosity tubes, of differing viscosity ranges, were placed in the bath. The heated bath meant that viscosity measurements could be carried out in the range 25°C-110°C.

2.14.2 Kinematic Viscosity Experimental Procedure

The experimental procedure was based upon the IP71[2.19] test method, with a few modifications due to the reduced sample size.

1. The heater bath was set to the desired temperature by adjusting the temperature controls and allowed to heat up. A accurate thermometer was placed in the bath, so that the fine temperature controls on the SetaVis could be adjusted to give the correct temperature.

2. Upon reaching the correct temperature the viscosity tubes were cleaned with petroleum ether (either 60-80, 80-100 or 100-120 depending on bath temperature). The external vacuum pump, attached to the SetaVis's viscosity tubes, drew the solvent down the viscosity tubes and was switched off when no visible traces of solvent were left in the sample tubes.

3. The oil sample was drawn into a 0.5ml glass syringe. The amount of oil drawn into the syringe depended upon which viscosity tube was being used, the range being 0.2-0.3ml. Care was taken to ensure no air bubbles were in the oil sample, because they would seriously impair the lubricant flow through the viscosity tube.

4. A viscosity tube was chosen[2] and the oil injected into its top. When the meniscus of the lubricant touched the first timing line a stopwatch was started. The stopwatch was stopped when the lubricants meniscus reached the second timing mark.

5. The viscosity tube was cleaned with petroleum ether and dried under vacuum. The same oil sample was then tested again (where sufficient samples was available), to give the average time of flow through the viscosity tube. If the two runs differed by more than ±2.5%[3] from the average then a further run was carried out.

The kinematic viscosity was calculated from the average measured time by using the following equation, represented by the units cSt[4].

$$v = T\,tc$$

where:-

v = kinematic viscosity
T = average time in seconds
tc = tube constant

[2] Care had to be taken to chose the 'correct' tube. Incorrectly chosen tubes gave false results by either allowing lubricant to flow through the tube or too quickly (tube too large) or by blocking the tube (tube too small).

[3] The repeatable value was obtained by measuring a sample of known viscosity twenty times to give the average viscosity. The standard deviation from the mean was ±2.5%, which was chosen as a guide to the repeatability of the test method.

[4] 1 cSt = $10^{-6} m^2 s^{-1}$

Viscosity measurements were carried out at 40°C and 70°C. Measurements were tried at 100°C but it was found that the ring zone samples produced air bubbles as the oil sample travelled down the viscosity tube. This was attributed to water and dissolved gas contamination, which was evaporating/desorbing in the tube, causing air bubbles. To calculate the Viscosity Index the kinematic viscosities at 40° and 100°C are needed. To obtain viscosity values at 100°C the 40°C and 70°C values were extrapolated to give an estimated value for 100°C using an algorithm supplied by a Petrochemical company.

2.14.3 Viscosity Index

Viscosity measurements are usually determined at more than one temperature because an oil's viscosity/temperature characteristics are very important. The change of viscosity with temperature depends on the oil type, so therefore an empirical scale known as viscosity index (VI), has been developed to indicate the change of viscosity with temperature. There are two ways to calculate viscosity index, shown below:

For VI's between 0 and 100

$$VI = \left(\frac{L - H}{L - U}\right) 100$$

where:-

U = the kinematic viscosity at 40°C of the oil whose VI is to be calculated.
L^5 = the kinematic viscosity at 40°C of an oil of 0 VI having the same kinematic viscosity at 100°C as the sample under test.
H^5 = the kinematic viscosity at 40°C of an oil of 100 VI having the same kinematic viscosity at 100°C as the sample under test.

For VI's above 100

$$VI = \frac{\left(\text{antilog}\left(\frac{\log H - \log U}{\log V}\right) - 1\right)}{0.0075} + 100$$

where:-

U = the kinematic viscosity at 40°C
V = the kinematic viscosity at 100°C
H = the kinematic at 40°C of an oil of 100 VI having the same kinematic viscosity at 100°C as the sample under test

[5] Both L and H are obtained from standard tables printed in the VI test, IP 226. Viscosity index is an arbitrary number calculated from the kinematic viscosity values at 40°C and 100°C and therefore has no units.

2.14.4 Factors Effecting Viscosity Changes

Many factors can effect a lubricant's viscosity, some of which are highlighted below:

An increase in viscosity can be caused by:-

- loss of base oil 'light ends' through partial evaporation,
- build-up of insoluble particulate matter,
- oxidation of the base oil, leading to crosslinked chains.

A decrease in viscosity can be caused by:-

- fuel contamination by a lighter hydrocarbon fraction,
- water contamination, introducing a dispersed second phase,
- shearing of the viscosity improver additive.

2.14.5 Kinematic Viscosity Results for CAT Oil

The viscosity results obtained show complex behaviour. The kinematic viscosity at 40°C, Fig. 2.12, show that for position 1 the viscosity is greater than the fresh oil. Positions 2, 3 and 4 show a decrease, with respect to the fresh lubricant.

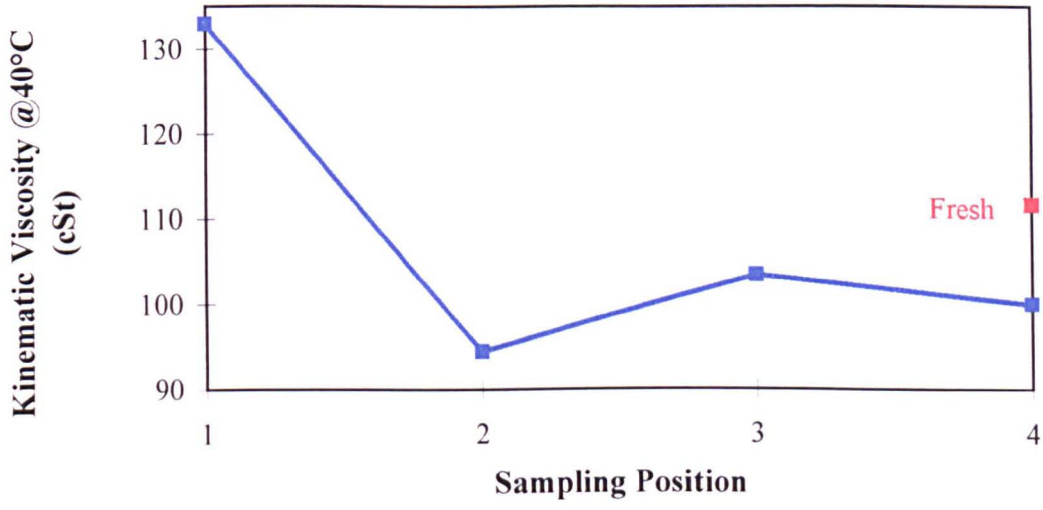

Fig 2.12 Comparison of Kinematic Viscosity at 40°C (CAT Oil) for Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

The calculated kinematic viscosity at 100°C also shows complex trends, Fig. 2.13. Positions 1, 2 and 4 show a slight increase compared to the fresh lubricant, however position 3 shows a large decrease.

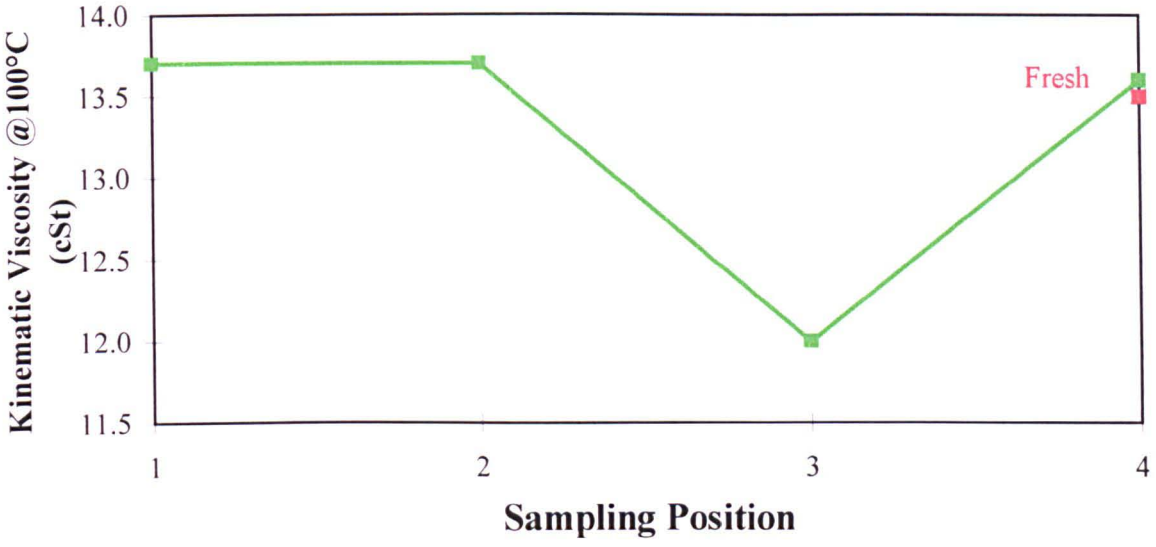

Fig 2.13 Comparison of Kinematic Viscosity at 100°C (CAT Oil) for Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

Preliminary investigations found that lubricant samples obtained from the Caterpillar 3406B were contaminated with small amounts of both water and fuel, which could have accounted for the viscosity decreases. However, it was thought, especially for position 3, that shearing of the viscosity improver additive may have occurred, which would also have accounted for the viscosity decrease. The increases in viscosity were thought to be due the evaporation of the 'light ends' of the base stock, which was investigated further, section 2.15.

2.14.6 Viscosity Index Results for CAT Oil

Viscosity index was calculated according to the IP 226[2.20] test method, section 2.14.3. From the obtained viscosity's the VI was calculated for the four sampling positions and the fresh oil, Fig. 2.14, for the CAT Oil.

The results show that for positions 1 and 3 the VI shows a decrease compared to the fresh oil, but an increase for position 2 and 4. Similar to the FT-IR C-O-C/sulphate formation findings, section 2.13.2, the VI results highlight the differences between the positioning of the sampling positions. Positions 1 and 3 are behind the second and top piston rings respectively and were subjected to different conditions, such as piston ring movement, which may have sheared the viscosity improver additive, leading to lower VI's. Positions 2 and 4 are on the face of the piston and therefore are not subjected to piston ring movement, which may lead to less shearing of the viscosity improver additive. The increase in VI was thought to be due the evaporation of the base oil, which was investigated in section 2.15.

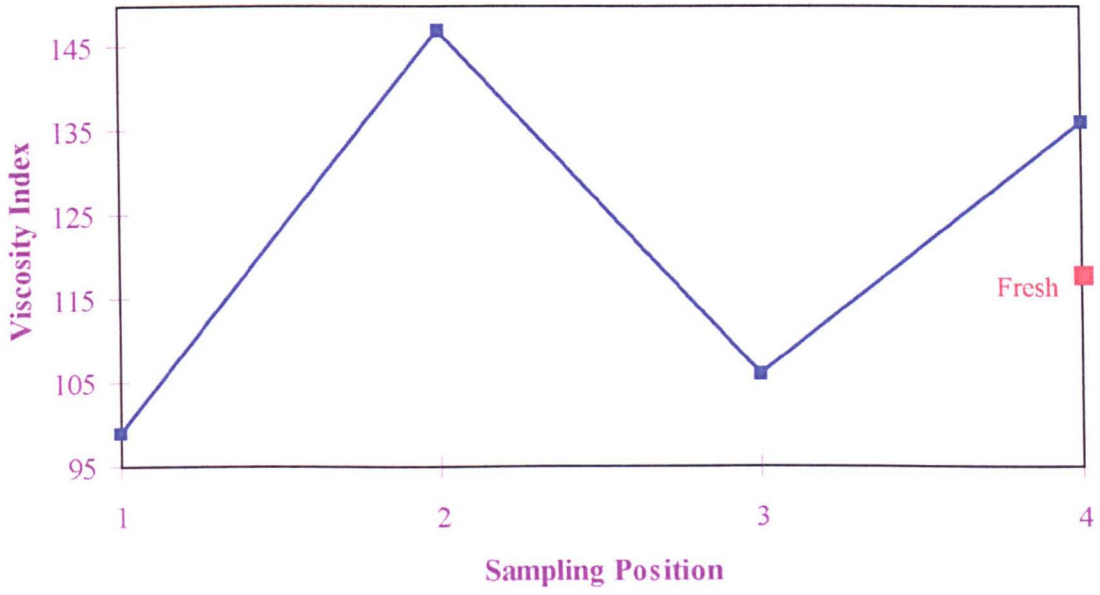

Fig 2.14 Comparison of Viscosity Index (CAT Oil) for Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

2.15 Thermogravimetric Analysis (TGA) to Measure Lubricant Volatility

Thermogravimetry is a technique in which the mass of a sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed and controlled. Thermogravimetric Analysis (TGA) was used to measure the volatility of lubricating oils during the course of this research. Volatility is an important lubricant property, due to the temperatures that lubricants can be subjected to, section 2.9, and also due to the wide spread of molecular weights found in lubricants. Lighter fractions, 'light

ends', of the lubricant can evaporate at these temperatures, causing increases in viscosity. Increases in viscosity can lead to lubricant pumping problems, especially upon cold starting.

2.15.1 Equipment

A Marlin Scientific TA51 TGA was used during the course of this work. Measurement of lubricant volatility was carried out according to the IP418[2.21] test method to find the relative volatility of automotive lubricants by isothermal thermogravimetry. The method involved placing a known mass of lubricant onto an aluminium pan, which was heated to a pre-set temperature and held at that temperature for a pre-set time. The mass loss over this time was then calculated using a computer programme. The lubricant mass loss was then related to the mass loss of the test standard, squalane (2,6,10,15,19,23-hexamethyltetraacosane)[4], to give the lubricant's relative volatility. The relative volatility value could be related to a common lubricant volatility test, known as the Noack test (DIN 51581).

2.15.2 Methodology for Volatility Measurement for Lubricants

The TA51 TGA consisted of a micro-electronic balance, a furnace capable of reaching 900°C and a personal computer containing the software which controlled the TA51 and the software used to calculate mass losses. A typical TGA test is described below:

1. The nitrogen gas flow rate was set at 80 ml min^{-1}. An aluminium pan[5] was placed onto the balance and tared.
2. The pan was removed from the balance and the oil carefully placed onto the upturned part of the aluminium pan using a piece of wire. The sample size used was 4.0mg ± 0.2mg. Care had to be taken to ensure that the lubricant did not overflow the upturned part of the pan.
3. The sample pan was placed back onto the balance and a method was selected for the machine to run.

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[4] Squalane was chosen as the standard because the volatility is similar to a typical motor oil, with a Noack value of approximately 10% weight loss.

[5] The aluminium pans used during the research were called SFI (Solid Fat Index) pans, supplied by Marlin Scientific. The pans had an upturned lip, onto which the sample was placed.

4. A typical method to measure the mass loss of a lubricant would involve equilibrating the temperature at 100°C, ramping the temperature at 10°C min^{-1} to a temperature between 195-205°C, where significant lubricant evaporation takers place, and then held at this temperature for 18 minutes.
5. After a test was completed the mass loss was calculated using the personal computer's software by measuring the mass loss between time X and time X + 15 minutes, where X[6] was the time the program took to reach the desired isothermal temperature.

To obtain the relative volatility the lubricants mass loss had to be related to the mass loss of the standard, which had been previously tested[7] , using the following equation.

$$RV = \frac{M_{sample}}{M_{squalane}}$$

Where:-

RV = relative volatility
M sample = mass loss of lubricant sample
M squalane = mass loss of standard

2.15.3 Volatility Results Obtained for CAT Oil

The TGA relative volatility results obtained for the CAT Oil show some interesting observations, which can also be related to the viscosity index results. Fig 2.15, surprisingly shows that for sampling positions 1, 2 and 3 the relative volatility was **greater** than the fresh lubricant. This increase in volatility was thought to be due to either:

- diesel fuel contaminating the lubricant sample. The 'heavy ends' of diesel fuel are very similar in carbon chain length to the 'light ends' of a lubricant. Therefore if diesel fuel contaminates the lubricant sample the TGA results can reflect this by showing an increase in volatility. Due to this similarity the detection and quantification of diesel fuel content in lubricants has proved difficult, although previous researchers have used TGA to detect and measure diesel contamination in lubricant samples[2.22].
- shearing of the lubricant in the piston ring zone, thus increasing volatility.

[6] Typical times were in the region of 15 to 18 minutes.
[7] The standard was run after every tenth sample.

The lubricant obtained from position 4 showed a **decrease** in volatility compared to the fresh lubricant, as would be expected. This result indicates that a portion of the 'light ends' of the lubricant had been lost through evaporation.

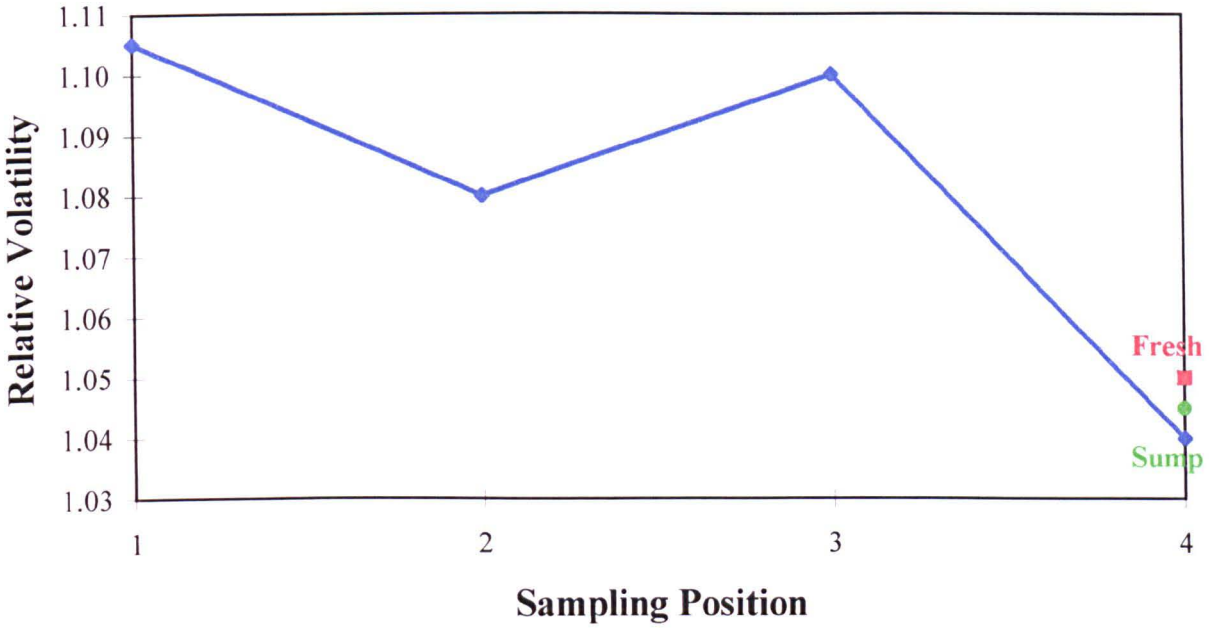

Fig 2.15 Comparison of TGA Results for CAT Oil Obtained from the Four Sampling Positions on the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

2.15.4 Comparison of Viscosity Index and Volatility Results

Examination of Fig 2.15 shows similar trends to Fig 2.14, the plot of VI over the four sampling positions. On combining the results, Fig 2.16, it can be seen that the VI results follow very similar trends to the volatility results, if the scale for the volatility was laterally inverted. The volatility decrease at position 4 corresponds to an increase in VI. This can possible be attributed to the loss of 'light ends' causing an increase in viscosity effecting the VI. It can also be argued that the loss of 'light ends' will increase the concentration of the VI additive, which could lead to the increase in VI. The increase in volatility, seen at positions 1, 2 and 3, corresponds to a decrease in VI.

This was thought to be due to either:

- diesel fuel dilution increasing the volatility, but decreasing the viscosity, which would effect the VI. Conversely to the statement above, diesel dilution would lower the concentration of the VI additive, possibly lowering the VI.
- shearing of the longer carbon chains of the lubricant into smaller fragments, thus increasing the volatility. If shearing had taken place then it was quite possible that shearing of the VI additive had also taken place, which would reduce the VI.

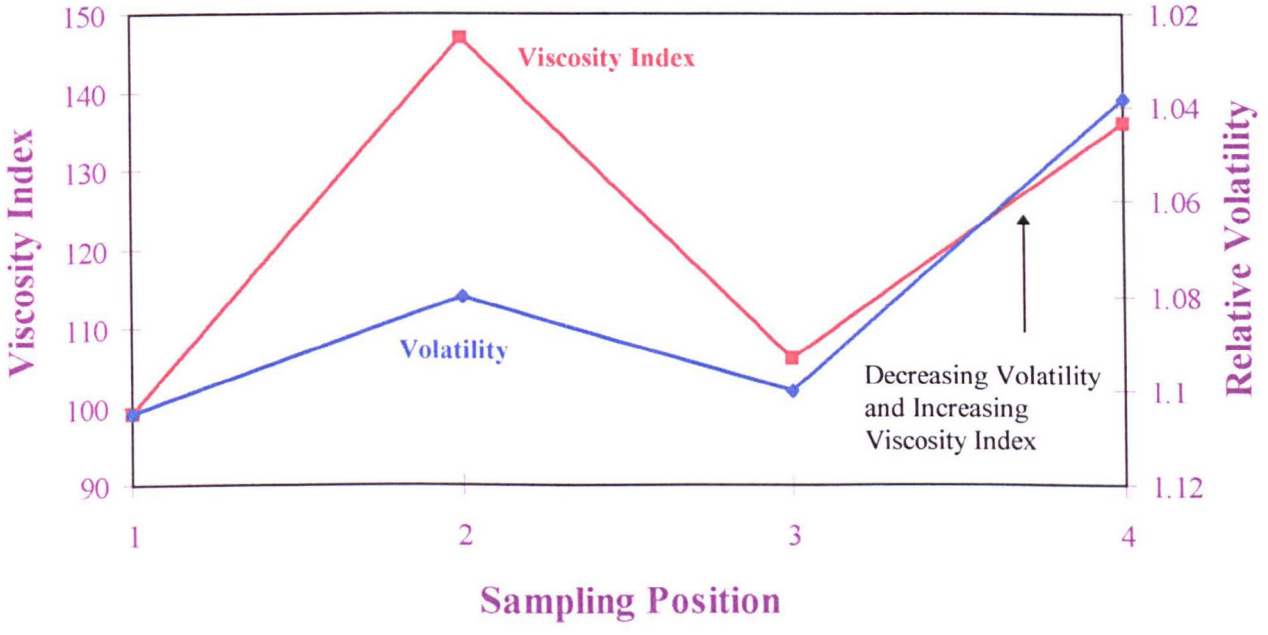

Fig. 2.16 Comparison of Viscosity Index and Volatility for the CAT Oil from Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

The changes in volatility noted highlights a potential weakness with respect to measuring viscosity and base number by FT-IR difference specta (ref: Section 2.13). It should be noted that allowances need to be made to correct for volatility changes to allow for absolute measurements of base number, otherwise measurements can be biased by the relative increase or decrease in the level of the additive level in the lubricant formulation. The limits of using spectroscopic measurement techniques to measure physical changes to a lubricant has been published[2.23], as has a way to compensate and correct for volatility changes[2.24].

2.15.5 Measured Metal Temperature and Volatility

Referring to the Templugs[R] results, section 2.9, it can be seen that the metal temperatures at positions 3 and 4 are above the temperatures where significant volatilisation takes place, therefore it is expected that significant oil consumption would be encountered. The particular TGA method used during the research gave mass losses in the order of 10% for synthetic based lubricants and 20% for mineral based lubricants, for temperatures in the order of 195-205°C. The metal temperatures at positions 3 and 4 are 198°C and 225°C respectively. It would therefore be expected to see decreases in volatility for samples obtained from these positions. However, referring to section 2.15.3, while position 4 showed a **decrease** in volatility, position 3 showed an **increase**. This demonstrates that the samples obtained had a propensity for being contaminated by diesel fuel.

2.16 Base Number

Lubricants are formulated, by the addition of basic additives (i.e. magnesium sulphonate, a detergent), to be basic in character so as to counteract acidic by-products of the combustion process. The measurement of this basic reserve, is therefore, an important indicator of lubricant performance and lubricant degradation. One such measuring technique is the Institute of Petroleum conductimetric test method, IP400[2.25], which is a non-aqueous titration method. Compared to others (i.e. potentimetric) this method has been shown by previous researchers[2.26, 2.27, 2.28, 2.29] to be particularly suitable for measuring the base number of ring zone samples because the end point of the titration is still clear, even with highly degraded and heavily soot laden samples, and less lubricant sample is needed for analysis.

2.16.1 Background

Base Number (BN) is defined as 'a quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, that is required to neutralise all basic constituents present in 1g of oil sample'. The unit of BN is therefore $mgKOHg^{-1}$. Typical gasoline lubricants are formulated to have an initial BN of 10 $mgKOHg^{-1}$, whilst diesel lubricants are typically formulated to have a BN of 15 $mgKOHg^{-1}$. These differences in BN

for gasoline and diesel lubricants are due to the nature of the fuels and their tendencies to form acidic products (i.e. gasoline fuel contains less 'acid forming' species than diesel fuel). High base numbers, such as the values seen in fresh lubricants, indicate that no strong acids are present. During the course of a lubricants working life the BN[8] tends towards zero, indicating the presence of strong acids. At a critical BN, which would be dependent on the application, the lubricant should be changed because it would be less likely to neutralise the acids generated.

2.16.2 Methodology

Analysis of the lubricant samples generated during the research were analysed using the IP400 test method. Prior to the analysis analytical grade chemicals were used to prepare the titration solvent (50% toluene, 45% propan-2-ol and 5% distilled water), and the acid (0.1M HCL in propan-2-ol). The BN apparatus consisted of a automatic burette, a magnetic stirrer, a platinum plate electrode, a balance capable of reading to three decimal places in grammes, a digital conductivity meter and a fixed volume dispenser for the titration solvent. The apparatus was interfaced to a personal computer, which was used to control the titration and log the data obtained. A typical BN analysis is described below:

1. 0.2-0.5g of oil sample was added into a pre-weighed sample beaker.
2. 20ml of titration solvent was added to the beaker which was sufficient to cover the electrode.
3. The apparatus was assembled and the stirrer switched on. Sufficient time was allowed for the oil to mix with the solvent.
4. The computer program was started, which controlled the titration. The acid was added to the solvent/oil mix in 50µl aliquots. A conductance reading was automatically taken when a steady conductance reading was obtained. The next aliquot was then added.
5. The conductance was logged by the program against volume of titrant added.
6. The program was either stopped manually once the end point had been reached, or stopped automatically after 2.5ml of titrant was added to the sample.

[8] A drop in BN should also be followed by an increase in Acid Number (AN). AN is a measure of the acidic products in the lubricant.

A typical conductance verses volume of titrant added plot can be seen in Fig. 2.17.

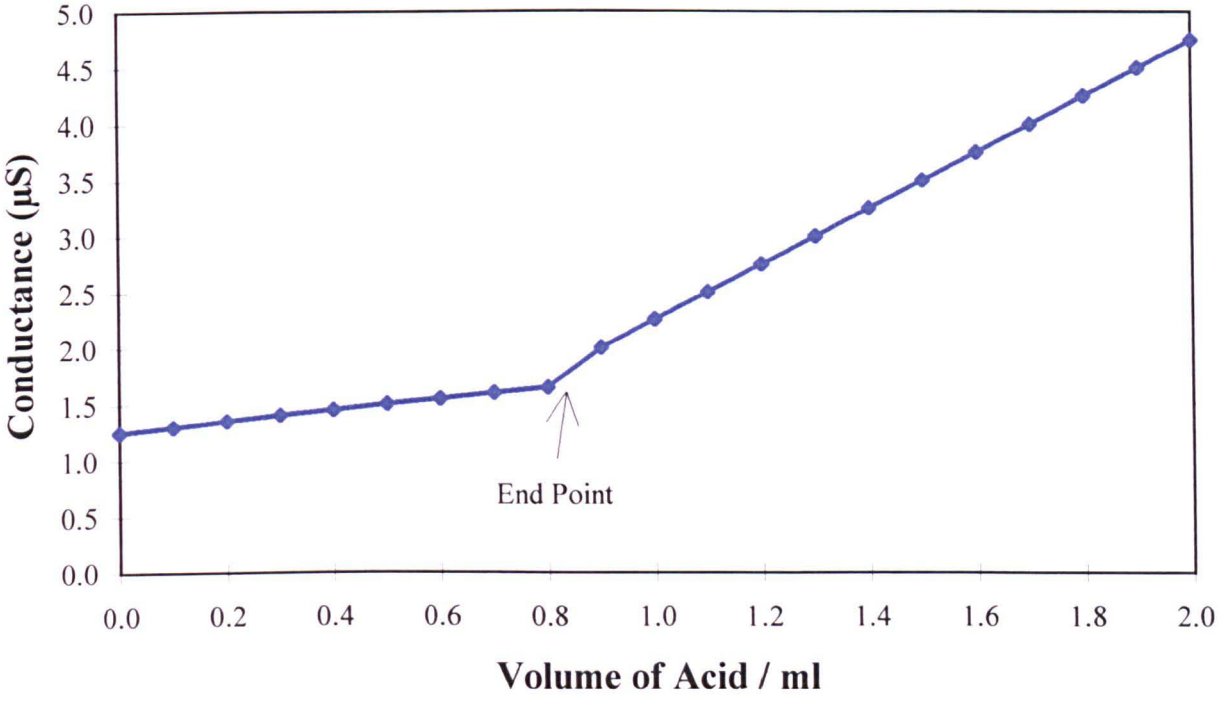

Fig. 2.17 A Typical Conductance Verses Volume of Acid Plot for a Ring Zone Sample Obtained from the Caterpillar 3406B Piston

The end point of the titration was the intercept of the two curves. The end point was calculated manually by printing out the titration curve and measuring the volume of acid added to reach the end point. To calculate the BN of the oil sample the following calculation was used.

$$BN = \frac{V\,M\,56.11}{m}$$

where:-

V = volume of acid added to end point
M = molarity of the acid used
56.11 = molar mass of KOH
m = mass of oil sample

2.16.3 Base Number Results for CAT Oil

The results for the CAT oil show a 20-50% decrease for the lubricant obtained from the four sampling positions, compared to the fresh lubricant, Fig 2.18. This showed that the oil was being significantly depleted of its basic reserve in the ring zone, although the decrease in BN was non-linear. The BN results also showed similar trends to the VI and volatility results. Although the BN decreased for all sampling positions, the decrease was lower for positions 2 and 4, which corresponded to the volatility **decrease** at these positions. It can therefore be argued that the basic additive concentration would have **increased**, which could possibly explain the higher BN's for these positions.

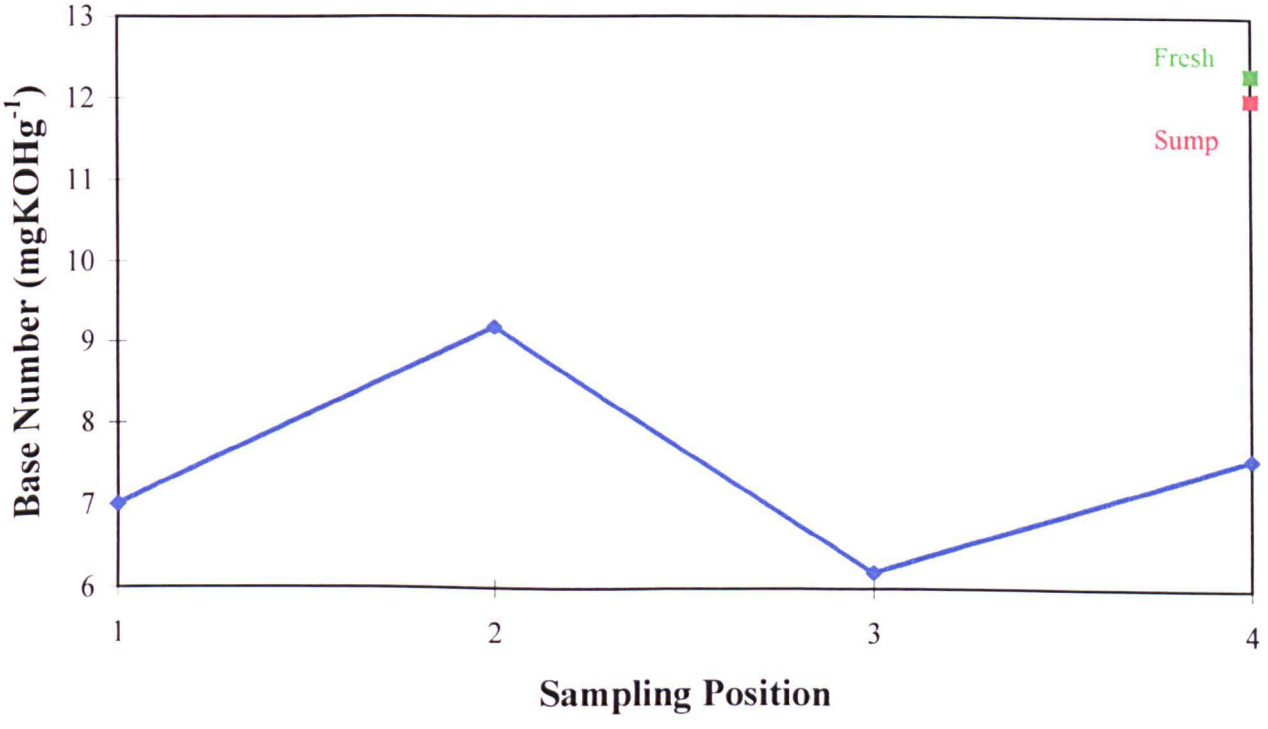

Fig. 2.18 Base Number Results for the CAT Oil from Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

2.17 Particle Size Analysis

Diesel particulates consist principally of combustion generated carbonaceous material (soot) onto which organic compounds may have been absorbed. Particulates are formed due to incomplete combustion of fuel, but can also be lubricant derived. The lubricant is designed to inhibit collation/deposition of these particles on metal surfaces through the action of detergent additives and also to keep these particles in suspension without agglomeration, achieved through the action of dispersant additives.

The measurement of particulate size in a lubricant is an important condition monitoring tool[2.26] because it indicates lubricant degradation and detergent/dispersant performance. Ineffective dispersant performance allows agglomeration of particles in cooler parts of the engine (i.e. the sump), which could lead to sludging and eventually to oil pumping problems. If agglomeration of particles took place in the piston ring zone possible problems could include piston ring sticking, which could eventually lead to breaking of the piston ring and possible engine seizure. This research concentrated on measuring particle size in the piston ring zone. One measuring device for measuring particle size is by laser light diffraction.

2.17.1 Background to the Laser Light Diffraction Method

Particle size measurements were carried out on a Malvern 2600 series laser diffraction particle sizer. The system was non-imaging, so the determination of particle size was based on the principle of laser diffraction.

The Malvern 2600 consists of a class 1 helium/neon laser, which passes through a beam expander to make the beam 1cm in diameter. This beam passes through the sample, where the laser light is diffracted according to the size of the particle, onto a detector. The detector consists of 31 concentric rings which measure the scattered light energy. Many detections are carried out over a few seconds, so the average scatter for the sample is found. The detectors provides an electronic output signal proportional to the light energy collected. This signal is processed by a computer where the data is subjected to statistical

analysis. The statistical analysis produces data which is related to the particle size distribution in the sample.

2.17.2 Methodology

The Malvern 2600 has a range of lenses of differing focal lengths, which allowed the detection of differing minimum and maximum detection limits in terms of particle size. For the initial experiments a focal length of 63cm was chosen, which gave detection limits between 1-118μm. Differing sampling applicators were available, with the small volume cell[9] chosen for the work due to the small amount of sample needed to obtain a reading. The cell is filled with a suitable dispersant (i.e. cyclohexane) to mix with the sample, and to stop total diffraction if the sample is too dark in colour. One of the implicit assumptions of the laser diffraction method is that the particles are spherical in shape. The small volume cell contained a magnetic stirrer, which agitated the sample/dispersant mix. Due to the many readings taken over a few seconds it was assumed that the particles were, in fact, spherical.

A typical experiment is shown below:

1. The small volume cell was filled with pre-filtered cyclohexane. The magnetic stirrer was switched on, and a background scan of the cell and the solvent was collected. The background[10] reading was subsequently subtracted from further analysis.
2. An oil sample was then added to the cyclohexane until the sample concentration was high enough to give a detectable reading[11] . The amount of oil added to the dispersant to achieve a measurable concentration was very dependant on the oil sample. Samples which contained a high concentration of particles (i.e. ring zone samples) needed only 0.1ml to reach the desired concentration, whilst sump samples needed up to 3ml to reach the desired concentration.

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[9] The small volume cell was made of aluminium and was fitted with two glass windows, through which the laser beam passed.

[10] A background reading was taken for each sample.

[11] A certain proportion of the laser beam had to be diffracted to achieve a good measurement. The amount of laser diffraction was related to the concentration of particles in the sample.

3. Particle size readings were then taken, and the results presented in tabular and graphical forms. The results were saved onto computer disk.
4. The cell was cleaned with cyclohexane ready for the next measurement.

2.17.3 Particle Size Distribution Results for CAT Oil

Visual inspection of the lubricant samples from the four sampling positions showed an increasing level of particulate matter as the sampling position progressed up the piston, which was represented in the particle size distribution results, Fig.2.19.

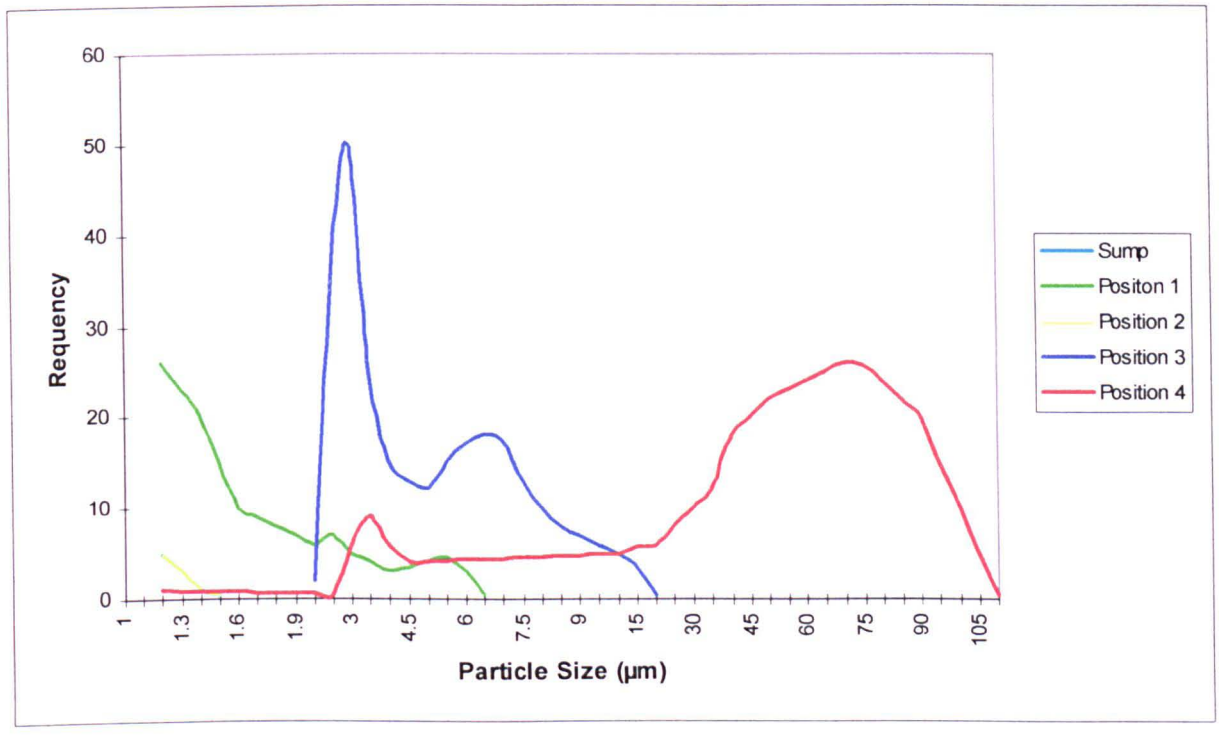


Fig. 2.19 Particulate Distribution of the CAT Oil for Samples Obtained from the Caterpillar 3406B Piston, Speed 750 rev min^{-1}, Load 89 BHP

It can be seen that the highest particle size distribution percentages for the various sampling positions were:

Sump = 1.2µm	
Position 1 = 1.2, 3, 5µm	Position 3 = 3, 7µm
Position 2 = 1.2µm	Position 4 = 3.5, 45, 80µm

Lubricant samples obtained from sample positions 3 and 4 showed particularly large particles, which are thought to be of importance in relation to the physical and chemical degradation of the lubricant. Close inspection of the sample collection vessels showed that the particulates were left on the side of the glass vial, whilst the oil remained at the bottom of the vial. The oil was golden in colour, but it was noticed that particles were suspended within it. This seemed to highlight a dispersant problem with this particular lubricant. If the dispersant additive had been performing correctly then it would have been expected that no large particles (i.e. over 2μm) would have been seen in the lubricant. Also, the oil sample would have been black in colour, because the dispersant would have been holding the particulates in suspension.

The results obtained suggest that the dispersant additive was not performing at sampling positions 3 and 4. Possible explanations for this are:

- due to the ring pack design there is very little lubricant in the ring zone of the Caterpillar 3406B, section 2.6.1. This layer of lubricant has to survive in extremely arduous conditions for long lengths of time, section 2.11.8. These conditions could be degrading the dispersant, (i.e. shearing)
- the dispersant is ineffective at the temperature associated at these sampling positions (i.e. at temperatures above 190°C)

2.18 MRI-1 Lubricant

The MRI-1 lubricant was developed for high temperature engine operation[2.30]. The work carried out on the lubricant during the project demonstrated that the MRI-1 lubricant behaved in a markedly different manner than the 'conventional' mineral, semi-synthetic and synthetic based lubricants used thoughout this project. Crucial points to note about the MRI-1 lubricant was that more lubricant was obtained from the sampling positions as the engine speed was **increased**, the opposite from the 'conventional' lubricants. One possible explanation for this was the exceptionally low volatility when compared to the conventional lubricants. The TGA test showed that the MRI-1lubricant 'lost' approximately 2-3% of mass, compared to 10 to 15% for the synthetic lubricants and 25 to 30% for the mineral

based lubricants. A visual representation of the low volatility characteristics of the MRI-1 lubricant was that the Caterpillar 3406B piston was extensively wetted by the MRI-1 lubricant after an engine test, which was not repeated with any other lubricant under test.

Changes in viscosity for the MRI-1 lubricant were also minor from lubricant samples taken from sampling position 3, when compared to the 'conventional' lubricants.

Two potential problems exist with the lubricant however. Firstly being that there was some evidence to suggest that the wear resistance properties of the lubricant may be less than ideal, which was noted by a Petrochemical company during an engine test. Secondly was that particle size analysis of MRI-1 samples from the various sampling positions showed similar trends to the CAT oil (ref: Section 2.17), which demonstrates that the MRI-1 lubricant may not be able to deal with high levels of insoluble matter.

In summary the MRI-1 lubricant was hardly effected by the conditions the Caterpillar 3406B subjected it to, whereas 'conventional' lubricants were substantially degraded. However care should be taken regarding the anti-wear and dispersant properties of this lubricant.

CHAPTER 3

Lubricant Transport Experiments

CHAPTER 3 Lubricant Transport Experiments

This Chapter describes in detail the lubricant transport experiments and results. The results obtained are compared to previous transport experiments, and developed into a lubricant flow model.

3.1 Introduction to Magnesium Tracer Experiments

Magnesium tracer experiments were carried out to:

- give a practical indication of lubricant transport through the ring pack of an operating diesel engine rather than theoretical models,[3.1, 3.2, 3.3, 3.4]
- to measure the time the lubricant took to transfer from the sump to the four sampling positions on the piston. These results would be related to previous research results[3.5], to see if any changes had occurred between lubricant transport times between 'older' and modern 'low emission' engines,
- using the lubricant transport results to develop a model of lubricant flow through the ring pack of an operating diesel engine, and relate these findings to oil consumption.

3.2 Magnesium Tracer Experiments using Oil F

Initial measurements of lubricant transport from the sump to sampling positions 1 and 3 were reported in Chapter 2, section 2.11. These initial experiments used CAT oil, a commercial SAE 15W/40 lubricant. Subsequent measurements of lubricant transport used oil formulation F, a SAE 15W/40 commercial lubricant. This lubricant was formulated with a calcium sulphonate based detergent, thus allowing a magnesium sulphonate based detergent to be used as the chemical marker.

Lubricant transport times were achieved for the four sampling positions using the same injection, sampling, measuring and correction techniques as have been reported previously (ref: Chapter 2 Section 2.11). The magnesium tracer results obtained are shown in Fig. 3.1. The measured appearance of magnesium as 'plugs' has already been commented upon in

Chapter 2. To obtain values from the results it was assumed that the first peak of magnesium that was measured was indicative of the arrival of magnesium at the sampling position. This was termed the 'residence time', or the time taken for the lubricant to travel from the sump to the piston sampling position. Using this assumption the results shown in Table 3.1 were obtained.

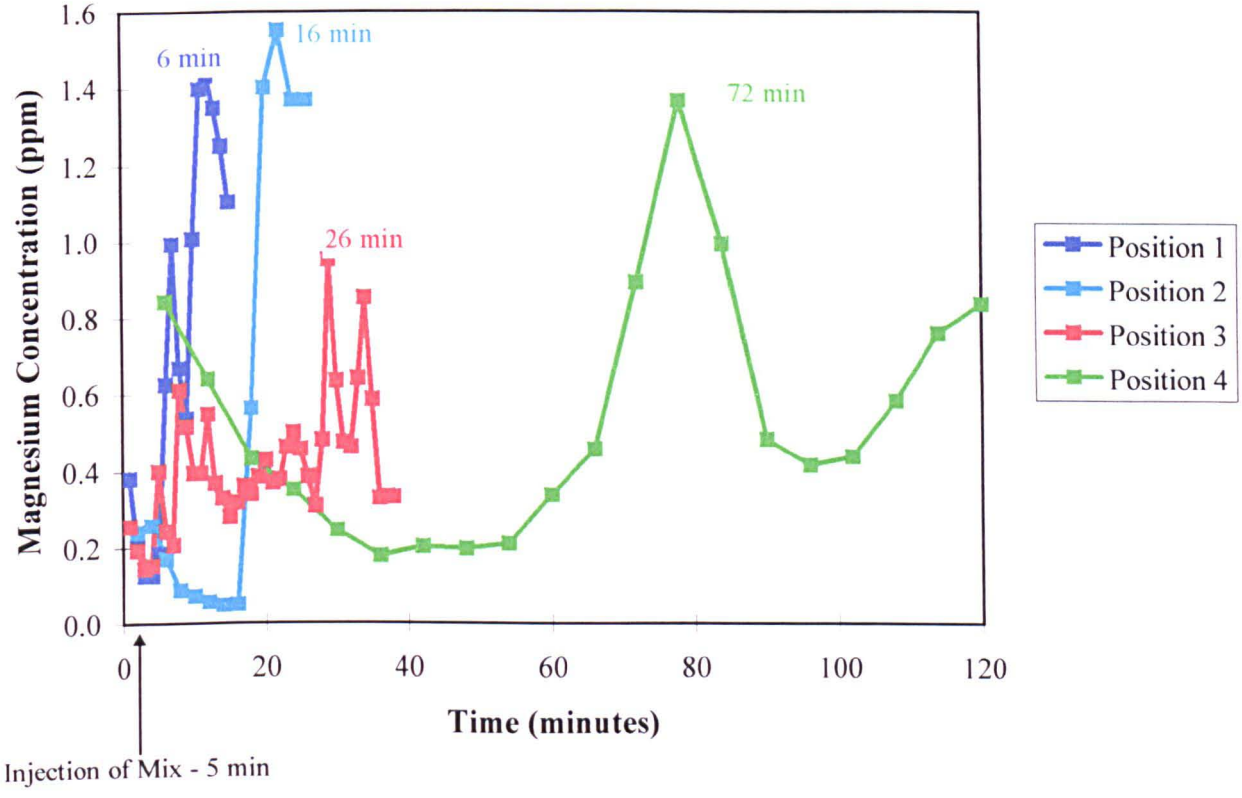


Fig. 3.1 Comparison of Lubricant Transport Rates for the Four Sampling Positions (SAE 15W/40 Lubricant - Caterpillar 3406B Engine, Speed 1500 rev min^{-1}, Load 130 BHP)

Sampling Position	Residence Time / minutes
1	6
2	16
3	26
4	72

Table 3.1 Residence Times for Lubricant Flow to the Four Sampling Positions

The residence times for lubricant transport to positions 1 and 3 using Oil F were similar to those obtained previously for the CAT Oil, 8 and 30 minutes, as reported in Chapter 2 section 2.11.8. Two possible reasons why these differences occurred could be attributed to, firstly differences in viscosity, although both lubricants were SAE 15W/40 grade, and secondly possible errors with the Magnesium injection and testing techniques.

Areas where errors could be introduced were:- the time taken to inject the magnesium sulphonate/oil mixture; differences in the chemistry of the CAT Oil and Oil F which could affect the lubricant flow (i.e. volatility); and analysis of the sample using ICP-AES. Although these errors were known, it was difficult to eliminate them. Therefore subsequent measurements were carried out using the same techniques, but with particular attention paid to the time the magnesium sulphonate mixture was injected into the sump.

The residence times obtained for Oil F showed that the lubricant was present in the ring zone of the Caterpillar 3406B for a considerable length of time, before it would be replenished with fresh lubricant. With reference to previous research, Cupples[3.6], reported that lubricant residence times for the Petter AA-1 and Caterpillar 1Y540 were between 3-5 minutes. Comparable times for the Caterpillar 3406B were found to be 26 minutes for Oil F and 30 minutes for the CAT oil. This large difference in residence times highlighted that, along with the **reduction** of lubricant present in the ring zone, the **residence** time had also increased significantly. This has shown a major difference between 'older' engines with a 'looser' ring pack, and a modern 'low emission' engine with a 'tighter' ring pack. It can therefore be shown that in a modern 'low emission' engine the lubricant has to perform under the same arduous conditions, but with thinner oil films, and for longer periods. This could eventually lead to wear problems at TDC, especially if the lubricant was not of a 'good' quality (i.e. a mineral base stock with poor viscosity and volatility characteristics). It can also be argued that these conditions are placing unacceptable stresses on a mineral based lubricant, and therefore a semi-synthetic or synthetic based lubricant should be recommended, with their inherently better viscosity and volatility characteristics.

3.2.1 Appearance of Magnesium at the Sampling Positions

As can be seen from Fig 3.1 the appearance of magnesium at the sampling positions was as a 'plug' and not as a sigmoidal curve, as had been previously expected. The appearance of peaks of magnesium had been termed 'plug flow', described in Chapter 2 section 2.11.8. As has been stated previously, the injected lubricant/magnesium mixture was thought not to be mixing in the sump, because the injection point chosen was just below the oil intake pump.

The continued appearance of 'plugs' of magnesium appearing seemed to suggest that either:

1. the lubricant/magnesium mixture was transported, as a 'plug', into the piston ring zone area, where it was sampled. The mixture was then transported back down into the sump, where it was drawn into the oil pump again, and the process repeated.

2. that the first 'plug' of measured magnesium was the mixture as it was transported past the sampling positions. Subsequent measured peaks were the mixture being re-sampled as it returned to the sump.

Point 1 seems unlikely due to the fact that it would take a considerable length of time for this to happen. It would, therefore, be expected that large time differences would be seen between the plugs, which was not the case. Subsequent 'plugs' would also have had smaller concentrations because the mixture would have been dissipated, to some extent, in the sump, however this was also not the case. It is therefore more likely that Point 2 was the reason numerous 'plugs' of magnesium were measured. This somewhat complicates the results, because it is very difficult to distinguish between a 'first pass' magnesium peak and a 'returning' magnesium peak. It was therefore thought reasonable to call the first magnesium peak the residence time, because subsequent peaks were probably lubricant returning to the sump from other sampling positions.

3.3 Comparison of Residence Times to Sampling Position on the Piston

Another representation of the magnesium tracer results is shown in Fig. 3.2. This figure plots the residence times compared to the distance the sampling positions were from the piston crown.

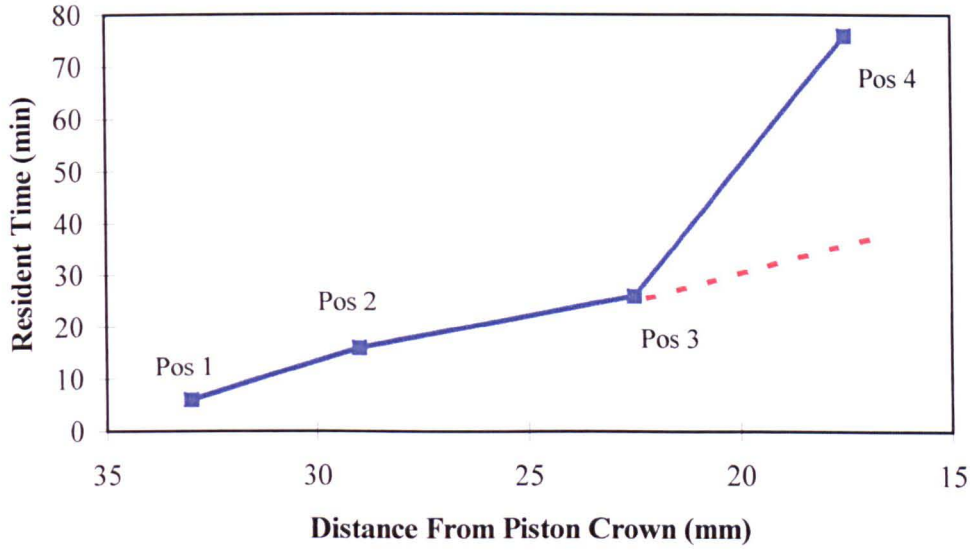


Fig. 3.2 Comparison of Residence Time as a Function of Sampling Position from the Piston Crown (SAE 15W/40 Lubricant - Caterpillar 3406B Engine, Speed 1500 rev min^{-1}, Load 130 BHP)

From Fig.3.2, it can be seen that the lubricant transport between sampling positions 1, 2 and 3 was almost linear, when compared to the distance the sampling position was from the crown of the piston. This highlighted that lubricant flow was **consistent** through the ring pack. The lubricant transport time to position 4, however, was much greater than the estimated time (dotted) that would be achieved if linear lubricant flow continued. This diversion from linear flow suggested that the top ring may have also been acting as a secondary oil control ring, thus inhibiting lubricant flow. If this was the case then this would reduce lubricant related emissions, by allowing less lubricant to pass the top ring, and thus enter the combustion chamber. However, this could possibly increase cylinder liner wear at TDC, especially if the lubricant was of a 'poor' quality.

3.4 Recycling of Lubricant in the Piston Ring Zone

A possible example of Point 2, referred to in section 3.2.1, can be seen in Fig. 3.4, which was measured at sampling position 2. It should be remembered that Position 2 is between the two piston rings. As can been seen, a peak was measured at 6 minutes after injection, another at 12 minutes, another at 18 minutes, with a much larger peak at 29 minutes. A possible explanation for this could be the recycling of lubricant within the piston ring pack. Previous research[3.7, 3.7, 3.9] suggests that there are two separate loops of recycling lubricant, a major one that passes through the relief holes under the oil control ring, and a minor one within the piston ring pack, Fig.3.3. It can be argued that Fig. 3.4 highlights the recycling of lubricant in the piston ring pack, with both lubricant being transported from position 1 and returning from position 3, being sampled at position 2. It should be noted that there is a regularity in the timing of the peaks which would suggests at 'pattern' to the recycling motion of the lubricant within the ring pack.

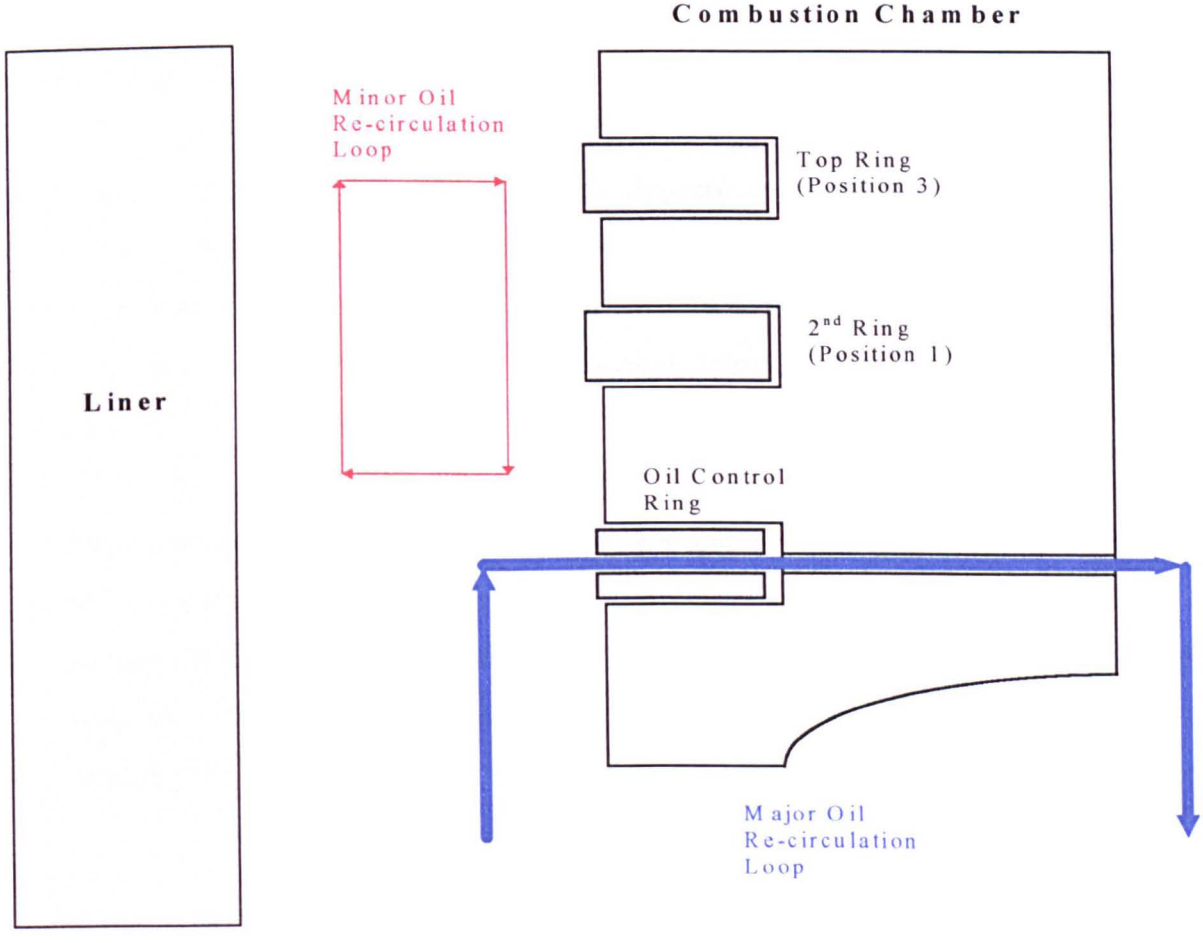


Fig. 3.3 Schematic Diagram showing two Possible Areas of Lubricant Recycling

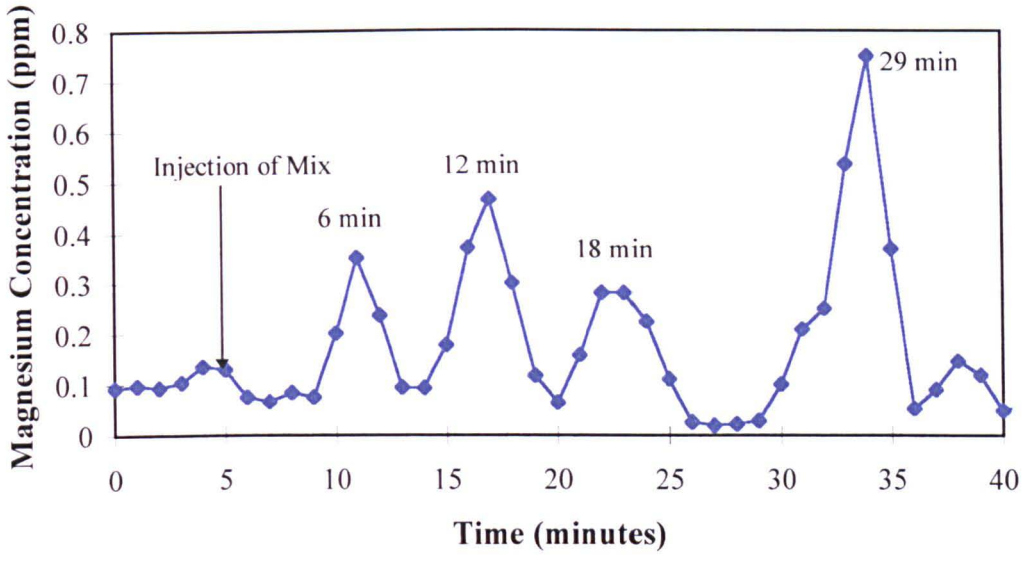


Fig. 3.4 Magnesium Concentration at Sampling Position 2, Possibly Showing Recycling of Lubricant in the Piston Ring Zone (SAE 15W/40 Lubricant - Caterpillar 3406B Engine, Speed 1500 rev min^{-1}, Load 130 BHP)

3.5 Investigation of Temperature of Sump Oil

As a result of magnesium tracer work, and the appearance of magnesium as 'plugs', it was decided to investigate the nature of the lubricant in the sump of the Caterpillar 3406B. It had been assumed that the lubricant in the sump would be subjected to violent mixing. However the occurrence of magnesium appearing as 'plugs' seemed to suggest that this was not the case.

To investigate the nature of the lubricant in the sump, a series of thermocouples were placed at strategic points within the sump. It was found that there was up to 8°C temperature difference within the sump oil. This highlighted a degree of stratification within the sump oil[9] , showing that mixing in the sump was not as 'violent' as had been assumed, and went some way in explaining the appearance of magnesium as 'plugs'. The work also

[9] It should be noted that the stratification would only be seen with engines on test beds. It would be unlikely that a vehicle on the road, with inherent vibrations from the road surface, showing a similar observation.

showed that engine flushes (when changing the test lubricant) should be extended from 15 minutes to at least 45 minutes to allow a complete turnover of the sump contents.

3.6 Lubricant Flow Model

From the residence times reported in section 3.2, it was possible to create a model of lubricant transport in the piston ring pack of the Caterpillar 3406B. This model gave an estimation of lubricant flow through the ring pack and the amount of lubricant that returned to the sump from the various sampling positions. The residence times were converted into a model of lubricant flow within the piston ring pack of the Caterpillar 3406B, by taking into account the distance the lubricant had to flow along the piston. Fig. 3.5 represents a schematic diagram of the lubricant model, with the piston inverted in the bore, which shows lubricant flow in the piston ring zone.

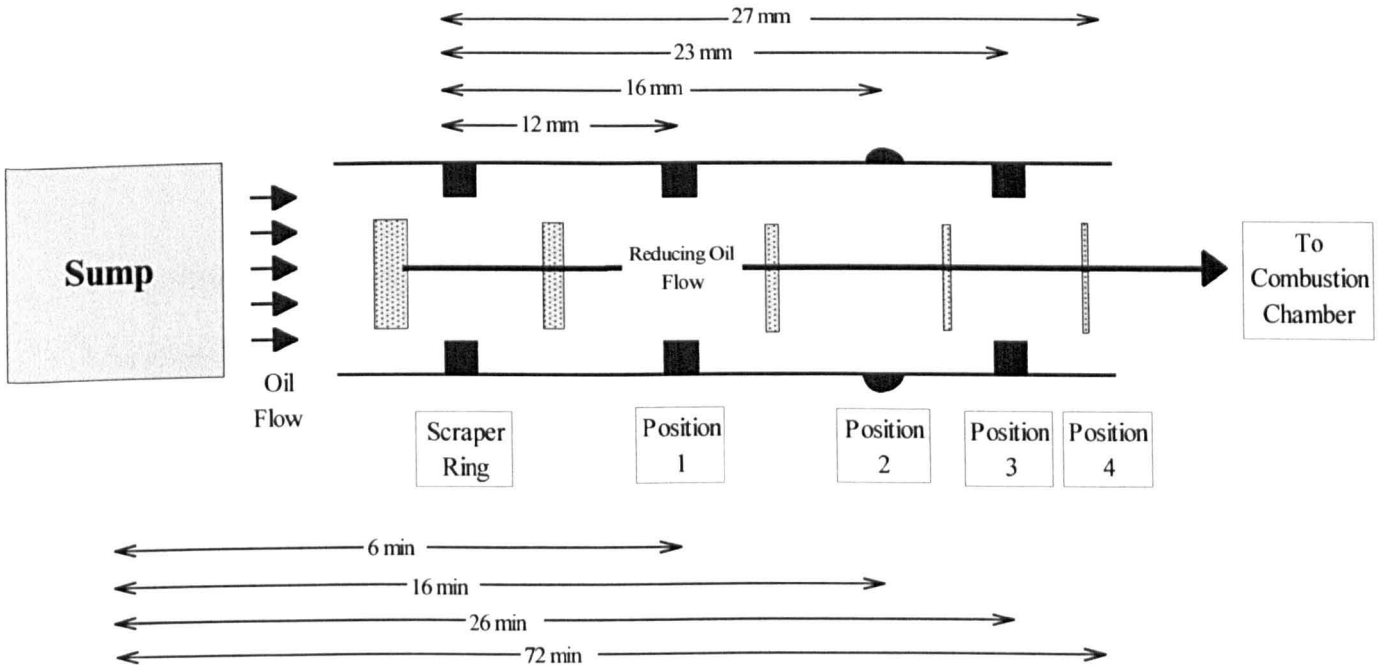


Fig. 3.5 A Representation of the Lubricant Flow Model through the piston Ring Pack of the Caterpillar 3406B

3.6.1 Explanation of Model

Assuming that the lubricant transport from the sump to the scraper ring is instantaneous (i.e. zero minutes). The velocity of the lubricant flow, v, is dependant upon the time it takes to travel a certain distance, d.

Equation 1

Therefore :- $v = \frac{d}{t}$

Where:-

v = velocity, in ms^{-1}
d = distance, in m
t = time, in s

Also, assuming the area of cross section between the piston rings and the cylinder bore is constant at a, another equation can be derived.

Equation 2

Therefore:- $Q = v\,a$

Where:-

Q = flow rate, in m^3s^{-1}
v = velocity, in ms^{-1}
a = cross sectional area, in m^2

On combining equations 1 and 2 we get:-

Equation 3

$$\frac{Q}{a} = \frac{d}{t}$$

Relating equation 3 to the measured values for magnesium transfer to the four sampling positions and their distances from the oil scraper ring, the following equations were deduced:-

For position 1 $\frac{Q_1}{a} = \frac{12 \times 10^{-3}}{360}$

For position 2 $\frac{Q_2}{a} = \frac{16 \times 10^{-3}}{960}$

For position 3 $\frac{Q_3}{a} = \frac{23 \times 10^{-3}}{1560}$

For position 4 $\frac{Q_4}{a} = \frac{27 \times 10^{-3}}{4320}$

(Where Q_x is the flow rate at each sampling position)

Assuming a is constant, these equations can be related to one another, in the form of simple ratios, to give lubricant flow rates from one sampling positions to another.

Therefore:-

For lubricant flow from position 1 to 2, it can be shown.

For position 1 $a = \dfrac{Q_1 \,.\, 360}{12 \times 10^{-3}}$ For position 2 $a = \dfrac{Q_2 \,.\, 960}{16 \times 10^{-3}}$

Combining these two equations gives:-

$$\frac{Q_2}{Q_1} = \frac{360}{960} \cdot \frac{16 \times 10^{-3}}{12 \times 10^{-3}} = 0.50$$

Which shows that **50%** of the lubricant that reaches position 1 is transferred to position 2.

Similar equations can also be formed for the other sampling positions.

For position 1 to position 3:-

$$\frac{Q_3}{Q_1} = \frac{360}{1560} \cdot \frac{23 \times 10^{-3}}{12 \times 10^{-3}} = 0.44$$

Which shows that **44%** of the lubricant that reaches position 1 is transferred to position 3.

For position 1 to position 4:-

$$\frac{Q_4}{Q_1} = \frac{360}{4320} \cdot \frac{27 \times 10^{-3}}{12 \times 10^{-3}} = 0.19$$

Which shows that **19%** of the lubricant that reaches position 1 is transferred to position 4.

The lubricant flow model showed that, of the lubricant that reached position 1, 19% of it was transferred to position 4. It would be expected that a high proportion of this lubricant would enter the combustion chamber. Relating these results to lubricant related emissions shows the importance of the oil control ring in reducing lubricant related emissions, by reducing the amount of lubricant that enters the piston ring pack. The model results have shown that to reduce lubricant emissions the amount of lubricant reaching position 1 (2^{nd} piston ring) must be reduced, especially if up to 19% of the lubricant that reaches this position could possibly be transferred into the combustion chamber.

Using similar equations to those already described, the amount of lubricant transported between each sampling position could also be calculated, shown in Table 3.2.

Sampling Position	Percentage Lubricant Transport	Returning Lubricant to the Sump
From Position 1 to Position 2	50%	50%
From Position 2 to Position 3	88%	12%
From Position 3 to Position 4	42%	58%

Table 3.2 Comparison of Lubricant Transport between Piston Sampling Positions and the amount of Lubricant that will Return to the Sump

Table 3.2 highlights some interesting points. The importance of piston rings, acting as a way of controlling oil flow, are clearly shown. The transport of lubricant from the piston rings (position 1 and 3) was far less than at position 2, which had no piston ring. This raised an important point regarding using the top piston ring as a secondary oil control ring, which would greatly inhibit the flow of lubricant into the combustion chamber. Therefore, the flow of lubricant from position 3 to 4, should be kept to a minimum, without increasing the chances of engine wear at TDC. It can be argued that the top piston ring (position 3) in these experiments was acting as a better oil control ring than the 2nd piston ring (position 1) because it allowed 8% less lubricant to be transferred to the next sampling position.

Table 3.2 also highlighted the fact that a considerable proportion of the lubricant that reached the piston sampling positions is transported back to the sump. This could explain the continued appearance of 'plugs' of magnesium seen as in Fig. 3.1 and Fig. 3.2, because the lubricant was transported back the sump, and consequently being sampled at other sampling positions.

3.7 Magnesium Tracer Experiment using a Sump Mixing Technique

Because the magnesium sulphonate/lubricant mixture injection technique was thought to be a possible cause for the occurrence of 'plug flow' an alternative injection technique was sought. It was decided to use technique 2, as described in Chapter 2 section 2.11.4, where

the engine was allowed to reach operating temperature, and then the entire sump contents were drained into an oil barrel. The magnesium additive was then added, and the barrel thoroughly shaken. The contents of the barrel was then placed back into the engine and the engine restarted, with samples taken immediately from the various sampling positions. This injection technique was termed 'sump mix'.

The tracer experiments were carried out as previously described, in Chapter 2 section 2.11, to measure the magnesium concentration. Results for the 'sump mix' experiments are shown in Fig. 3.6. The results showed that the time to reach the maximum magnesium concentration after injection for position 1 was 10 minutes. This showed a 4 minute increase from the 'plug flow' technique, at 6 minutes. However, for position 4 the time was 45 minutes, showing a large decrease from the 'plug flow' value of 72 minutes.

This work produced conflicting results. By the nature of the injection technique a reduction in residence time was expected. This expected reduction in residence time would have been brought about by the engine being stopped and the lubricant drained, which would allow the engine to cool. Upon restarting, the engine would no longer be at operating temperature. Thus the piston would not be fully expanded in the cylinder, appearing 'loose'. This would lead to an increased amount of lubricant being present in the ring pack from start up, until the normal operating temperature of the engine was obtained. It was thought that this increased lubricant flow would reduced the residence time. This was the case for position 4, but the opposite occurred for position 1. This occurrence was very difficult to explain, but could be related to the continued appearance of the magnesium as 'plugs'.

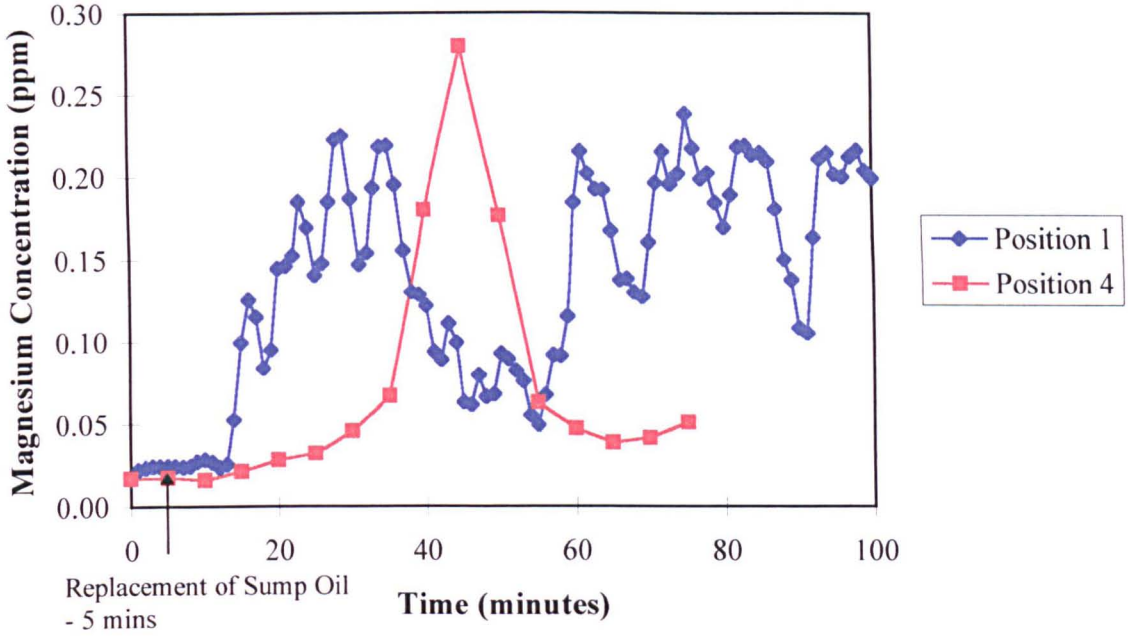


Fig. 3.6 Lubricant Transport Rates for Piston Sampling Positions 1 and 4 using the 'Sump Mix' Injection Technique (SAE 15W/40 Lubricant - Caterpillar 3406B Engine, Speed 1500 rev min^{-1}, Load 130 BHP)

3.7.1 Results from the 'Sump Mix' Experiments

The appearance of the graphs obtained from the 'sump mix' experiments was very surprising. Once again the graphs appeared to show 'plug flow'. It was known that the sump oil was thoroughly mixed with the magnesium additive external from the engine, therefore a uniform concentration of magnesium would have been in the sump oil. This should have produced a sigmoidal curve of measured magnesium, as had originally been expected. Three possible explanations for the continued appearance of magnesium as 'plugs' using the 'sump mix' injection technique are highlighted below:

1. the concentration of the magnesium additive effected the delicate balance of the lubricant base oil/additive pack. This could have caused the magnesium to 'drop out' of suspension, producing a non-uniform mixture,

2. the temperature that the additive was subjected to in the piston ring area (166°-225°C) was causing the lubricant/magnesium additive mixture drop out of suspension, producing a non-uniform mixture,

3. the detergency effect of the magnesium additive means that it 'cleans' metal surfaces. This could mean that carbon particulates were coated with the magnesium additive. These particles would be sampled by the ring zone sampling system, leading to a sample containing liquid oil with a low magnesium concentration and carbon particulates of a far higher magnesium concentration. When the samples were tested for magnesium concentration using the ICP-AES technique, it could not be ascertained whether the sample had a uniform magnesium concentration. i.e. could a carbon particle (containing a large magnesium concentration) have passed through at a crucial moment in the measuring cycle, thus producing a falsely high magnesium concentration.

It was though unlikely that point 3 was the cause for the continuing appearance of magnesium as 'plugs' because of the regularity of their appearance, refer to Fig. 3.1 and 3.4. Also, due to the nature of the sampling positions, more carbon particulates were obtained at positions 3 and 4, therefore it would be expected to see more 'peaks' at these positions, which was not the case. Therefore the most likely reason was that the magnesium additive had dropped out of suspension, due to either point 1 and/or point 2. Referring to section 3.7, point 1 seems the most likely explanation.

3.8 Measurement of the Magnesium Content in the Sump Oil

During the 'sump mix' experiments the magnesium concentration in the sump oil was investigated, by removing samples at 5 minute intervals. These samples were removed via a drain pipe which had been tapped into the bottom of the sump. A small amount of sample was allowed to drain to waste, before lubricant was collected in the sample vessel. These samples were analysed and presented in the same format as has been described earlier. The sump results can be seen in Fig. 3.7, which shows the concentration of magnesium in the sump, compared to those found at positions 1 and 4.

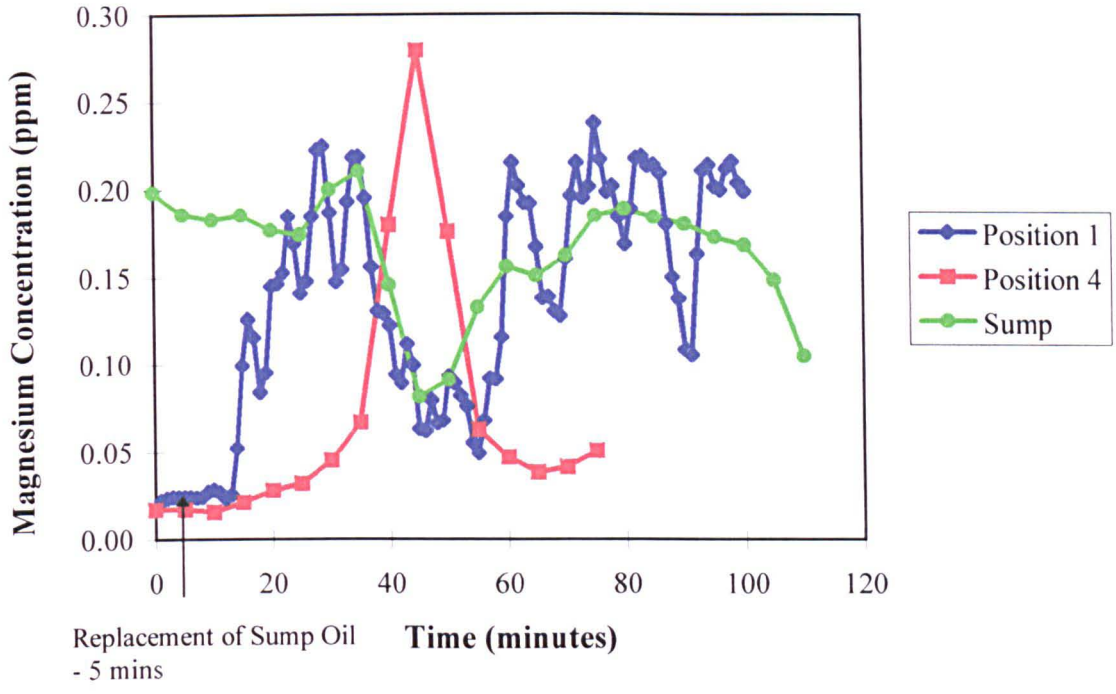


Fig. 3.7 Comparison of Magnesium Concentration, using the 'Sump Mix' Injection Technique, Obtained from Sampling Positions 1, 4 and the Sump (SAE 15W/40 Lubricant - Caterpillar 3406B Engine, Speed 1500 rev min^{-1}, Load 130 BHP)

The most striking results from the sump measurements was the reduction in magnesium concentration 30-40 minutes after injection. This also corresponded with a **decrease** in the magnesium level measured at position 1, and a **increase** in the magnesium level at position 4. As the magnesium level **decreased** at position 4 the level **increased** at position 1 and the sump. These results appear to show that:

- mixing was not very 'violent' in the sump, which leads to stratification,
- the magnesium additive was dropping out of suspension, causing 'plug flow' even though the contents of the sump oil had been thoroughly mixed with the magnesium additive,
- the 'plugs' of magnesium appeared to flow to position 4 at the same time, thus reducing the magnesium concentration at the sump and position 1. When the magnesium returned from position 4 the level of magnesium rose at position 1 and in the sump.

3.9 Conclusions from Magnesium Tracer Experiments

- The time the lubricant takes to travel from the sump to the top ring has risen dramatically (approximately 10 fold) between 'older' and modern 'low emission' diesel engines. This could lead to the further introduction of semi-synthetic and synthetic based lubricants, which are better suited to the demands that this places on the lubricant.

- The model of lubricant flow developed shows that 19% of the oil that reached the 2nd piston ring is transferred to the base of the crown land sampling position. It would be expected that a large proportion of this lubricant would enter the combustion chamber. Therefore the lubricant that reaches the 2nd piston ring must be kept to a minimum if lubricant related emissions are to be kept low. This highlights the importance of the oil control ring in reducing lubricant transport into the ring zone, and ultimately in reducing lubricant related emissions. This point also highlights that semi-synthetic and synthetic based lubricants are required, because oil volume in the piston ring zone has been reduced.

- The magnesium tracer technique showed the appearance of magnesium as 'plugs'. A possible explanation for this is the recycling of lubricant in the piston ring pack. This could lead to information on the rate of recycling, and how this is affected by differing ring pack configurations, and the rate of lubricant transported back to the sump.

- The tracer work has shown the sump was not as 'violent' a mixing environment as had been initially thought. A degree of stratification in the sump was noted, using temperature measurements and by monitoring magnesium concentration. This had implications with engine flushing periods, which were increased to ensure the complete turnover of the sump contents. This has a major implication for sequential lubricant tests in standard engine tests. This information has been applied to routine engine test situations, and has lead to an improvement in test reproducibility when the lubricant 'flush' times were extended from 15 minutes to 45 minutes.

CHAPTER 4

Statistical Analysis of Experimental Data

CHAPTER 4 Statistical Analysis of Experimental Data

This Chapter presents a statistical analysis of the analytical data obtained from this study. Using common statistical techniques the lubricant degradation is related to lubricant type, engine speed, fuel sulphur content, piston ring zone sampling position and piston ring pack type.

4.1 Testing Matrix

During the course of the research project specific lubricants were subjected to a matrix of engine running conditions in the CAT 3406B. This matrix consisted of running the CAT 3406B at various engine speeds (typically 750, 1100, 1200, 1300, 1400, 1500 and 1800 rpm), sampling lubricant from various sampling positions (typically the sump, position 1, position 2, position 3, and position 4), using piston rings packs of different designs (pre-1991, 1991 and 1994 US emission control ring packs) and fuels of different sulphur content (1%, 0.3%, 0.18%, 0.05%, 0.042% and 0.009%).

All lubricants sampled during the testing matrix were subjected to a suite of analytical tests to assess changes, or degradation, of the lubricant in relation to sampling position, engine speed, fuel sulphur content and piston ring pack type. The suite of analytical tests typically employed were ThermoGravimetric Analysis (TGA), Base Number (BN), Kinematic Viscosity and Fourier Transform Infra Red (FTIR). These were chosen to give a insight into key chemical and physical changes to the lubricant, and because they are routinely used in lubricant quality control laboratories. These tests could be able to assess lubricant quality and lubricant degradation in relation to the engine testing matrix described above.

4.1.1 Analytical Testing

All analytical tests were carried out using the methods described in Chapter 2, Section 2.13 to 2.15. A database was used to record the engine speed used, fuel type, piston ring type and sampling position from where the lubricant sample was obtained. This database was also used to record the analytical tests carried out on the lubricant.

Due to the nature of piston ring zone sampling on the CAT 3406B it was noted that under certain engine running conditions and settings very little lubricant was obtained from the sampling positions (especially position 3 and position 4). As the research project evolved it was decided that engine runs would be limited to either 10 hours or when a sample size of approximately 1g was obtained. Due to this constraint some lubricant samples obtained were too small in size to carry out the complete suite of analytical tests on it (note: approximately 1g of lubricant was needed to complete all the analytical testing). Therefore a flow chart was developed which showed the order of importance which was placed on each analytical test. This is shown in figure 4.1

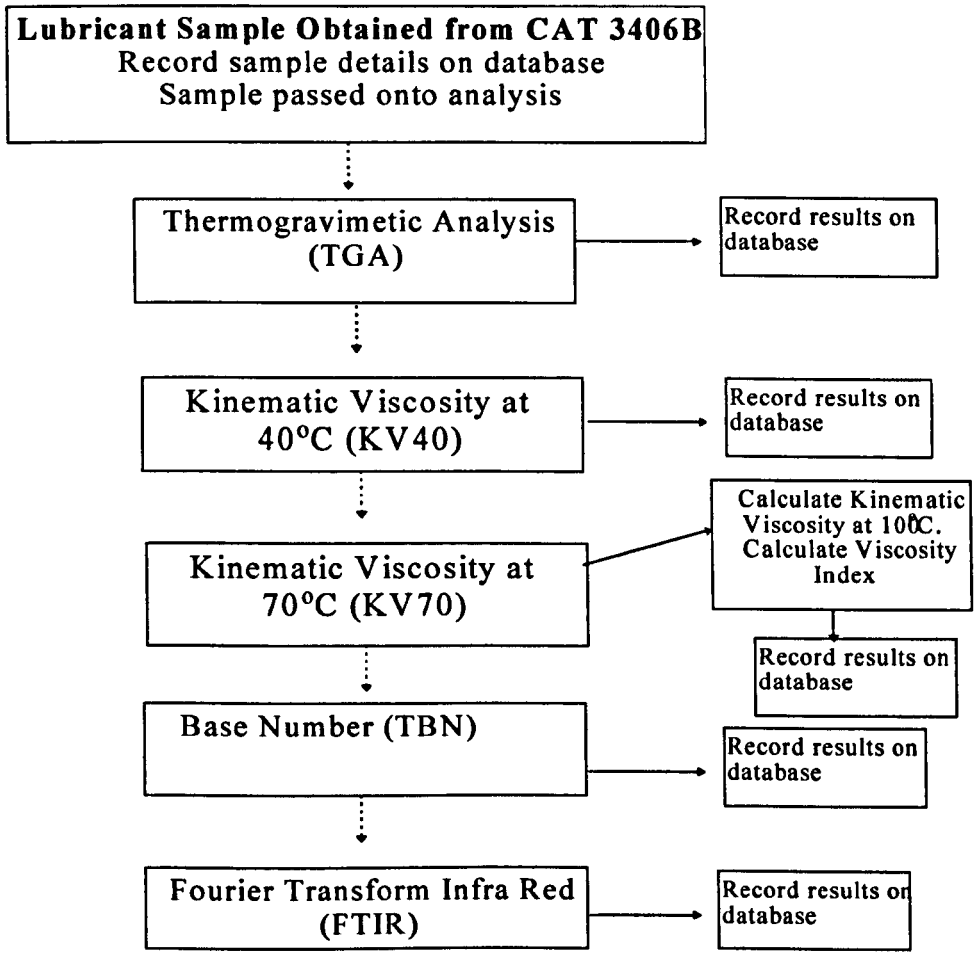


Fig 4.1 Analytical Testing Flow Chart Employed During Research Project

4.2 Analysis of Results

Due to the nature on the engine testing matrix and suite of analytical tests employed the results were complex. There were many measured variables (TGA, BN, Kinematic Viscosity, FTIR) carried out on the lubricants at various engine speeds and with various fuels and piston ring packs. To obtain the most from the data generated needed a statistical approach to the data analysis. Therefore the analytical results obtained were analysed by common statistical techniques which also enabled the engine variables (piston ring type, fuel sulphur level, sampling position and engine speed), where applicable, to be evaluated. The statistical techniques employed are discussed in Chapter 4, Section 4.4. The computer packages used for the statistical analysis were MS-Excel, MINITAB and SCAN.

4.3 Experimental Data

During the course of the research project the project evolved into three separate, but linked, pieces of work. These were subjected to the suite of analytical tests described in Chapter 4, Section 4.2. The three pieces of work are described below:-

1) Examination of the effects of engine speed on the degradation of various lubricant formulations.

- Lubricants used were CAT oil (a commercial lubricant), two mineral lubricants (Oil A & Oil B), one semi-synthetic lubricant (Oil C) and two synthetic lubricants (Oil D and Oil E).
- Engine speeds ranged from 750rpm to 1800rpm
- The diesel fuel used for all experiments was 1% sulphur reference fuel.
- The piston ring pack used were pre-1991 US Emission Control standard.
- Oil samples were taken from the sump, Position 1, Position 2, Position 3 and Position 4.

2) Examination of the effects of fuel sulphur content on the degradation of various lubricant formulations.

- Lubricants used were a mineral lubricant (Oil G), a semi-synthetic lubricant (Oil H) and a synthetic lubricant (Oil I).

- Engine speed was constant at 1300rpm
- The diesel fuel used for the experiments contained either 0.3% or 0.05% sulphur.
- The piston ring pack used were pre-1991 US Emission Control standard.
- Oil samples were taken from the sump, Position 1 and Position 3.

3) Examination of the effects of piston ring pack type and fuel sulphur content on the degradation of various lubricant formulations.

- Lubricants used were a mineral lubricant (Oil J), a semi-synthetic lubricant (Oil K) and a synthetic lubricant (Oil L).
- Engine speed was constant at 1300rpm
- The diesel fuel used for the experiments contained either 0.18%, 0.042% or 0.009% sulphur.
- The piston ring pack used were 1991 and 1994 US Emission Control standard.
- Oil samples were taken from the sump, Position 1 and Position 3.

Note: Further details on the lubricants used for the experiments are detailed in Chapter 2, Section 2.12. Further details on the sampling positions are detailed in Chapter 2, Section 2.4.

Due to the separate nature of the three pieces of experimental work it was not thought advisable to analyse the results as one complete dataset. The reason for this was that not every lubricant type was run using every variable (i.e. not every lubricant was run using the different fuel types or piston ring packs). Analysis of the all the data together would therefore have meant there would be omissions in the dataset, which would have clouded any analysis and conclusions. Therefore each piece of work was analysed separately, however the conclusions from each piece of work were considered before the next piece of work was undertaken.

4.4 Statistical Techniques Used to Analyse Data

As described in Chapter 4, Section 4.2, the analytical results obtained were subjected to commonly used statistical techniques. Previous research[4.1, 4.2, 4.3] has shown that statistical analysis is highly suited to lubricant analysis, due to the complex nature of the results and the number of variables measured (which are usually inter correlated).

The statistical analysis utilised during the project are detailed in Sections 4.4.1 to 4.4.5.

4.4.1 Descriptive Statistics

Descriptive statistics is a simple tabular output of the mean and standard deviation for the measured variables The technique can be used however to calculate the mean and standard deviation for different variables such as engine speed, fuel sulphur level and piston ring pack type.

The descriptive statistics give a general understanding of the data, which can then be applied to the more complex statistical techniques.

4.4.2 Principal Component Analysis (PCA)

PCA is a technique where the relationships between variables are analysed. The fundamental idea is to transform correlated variables into a new set of uncorrelated variables called Principal Components (PC's). These new variables (PC's) are linear combinations of the original variables, formed sequentially in such a way that the first PC accounts for as much variation in the data as possible. The second PC accounts for as much of the remaining variation, whilst being uncorrelated with the first PC.

Ideally the first few PC's will capture most of the variation in the data, thus reducing the number of dimensions in the dataset, without losing much information. The ideal scenario is to reduce the dimensions to two, so that a simple bivariate plot can be drawn.

Due to the number of variables measured as part of the project, and the potential correlation between these measured variables it was thought that using PCA was an ideal way to effectively reduce the number of dimensions in the dataset, therefore making interpretation of the data simpler.

The typical output from PCA is:-

- A tabulated representation of the PC's, which also contains a cumulative total of how much variation the PC's are describing.
- A Principal Component Loading Plot which plots the importance (Loadings) for each variable against the first and second PC's. This plot shows the presence of relationships between the original variables, highlights particular variables which are important or unimportant, and detail which variables are influencing the PC's.
- A Principal Component Score Plot which plots the second score matrix against the first score matrix. This plot is an object orientated summary of the data which can be used to detect relationships between the objects, highlight groups of objects or identify objects which are outlying.

4.4.3 Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) is similar to regression in that it is used to investigate and model the relationship between a response variable and one or more independent variables. However, analysis of variance differs from regression in two ways: the independent variables are qualitative (categorical), and no assumption is made about the nature of the relationship (i.e. the model does not include coefficients for variables). In effect, analysis of variance extends the two-sample t-test for testing the equality of two population means to a more general null hypothesis of comparing the equality of more than two means, versus them not all being equal.

ANOVA is the test of equality of the means of several groups (i.e. fuel type, piston ring type) in respect to a response variable (i.e. BN, TGA).

The ANOVA analysis used during the project was the General Linear Model (GLM) function in MINITAB. GLM fits the general linear model for univariate responses. In matrix form this model is Y = XB + E, where Y is the response vector, X contains the predictors, B contains parameters to be estimated, and E represents errors assumed to be normally distributed with mean vector 0 and variance S. Therefore it was implicitly assumed that the relationships between the measured variables and the factors were linear.

The GLM output contains the p-value for each factor measured against each variable. The p-value (also termed the observed significance level or probability level) indicates whether the factor being investigated (i.e. engine speed) is having a significant effect on the variable (i.e. BN). During this project a p-value of 0.05 or less has been considered to indicate a significant relationship. Therefore a p-value of 0.05 or less will indicate that the factor is effecting the measured variable by a linear relationship, whilst a p-value greater than 0.05 will indicate that the factor is not effecting the measured variable by a linear relationship.

4.4.4 Main Effects Plot

A main effects plot is a plot of means at each level of a factor (where the factor was either oil type, engine speed, fuel sulphur content, piston ring type or sampling position). The plots were conducted for all measured variables. The main effects plot displays the response mean (i.e. BN) at each factor. The points in the plot are the means at the various levels of each factor, with a reference line drawn at the grand mean of the response data. The vertical dotted lines represent the individual 95% confidence intervals for the mean of each factor. The effects noted are the differences between the means of the factors and the reference line.

4.4.5 Matrix Plot and Correlation Matrix

A matrix plot is a two-dimensional matrix of individual plots. Matrix plots are good for, among other things, seeing the two-variable relationships amongst a number of variables all at once, rather than plotting a series of bivariate plots.

The Correlation Matrix tabulates the correlation coefficients between the variables. The correlation coefficient lies between -1 to +1. If one variable tends to increase as the other decreases, the correlation coefficient is negative. Conversely, if the two variables tend to increase together the correlation coefficient is positive. A p-value is also displayed for the hypothesis test that the correlation is zero. A p-value of 0.05 (or less) has been used to indicate that the correlation coefficient is not zero. Note: if the coefficient of determination is required (R^2 value) then the correlation coefficient has to be squared.

4.4.6 Coding of Variables Used During Data Analysis

Due to the nature of the computer packages used it has been necessary to code the measured variables to allow data analysis to take place. The key in Table 4.1. details the code used and the variable name.

Code	Variable Name	Code	Variable Name
ZDDP P=S	ZDDP P=S depletion as measured by FTIR difference spectra	TGARV	Relative Volatility as measured by TGA
ZDDP POC	ZDDP POC depletion as measured by FTIR difference spectra	Visc40C	Kinematic Viscosity measured at 40°C
C Ox	Carbonyl Oxidation formation as measured by FTIR difference spectra	Visc100C	Kinematic Viscosity measured at 100°C
NE	Nitrate Ester formation as measured by FTIR difference spectra	VI	Viscosity Index
COC	COC oxidation formation as measured by FTIR difference spectra	BN	Base Number

Table 4.1 Key to Coding of Measured Variables

4.5 Analysis of Fresh Oil Samples

Initial analytical analysis was carried out on the fresh oil samples, as this gave an indication of similarities and differences between the lubricants. The samples were tested for volatility (expressed as relative volatility) by TGA, Kinematic viscosity and Base Number (BN).

These analytical techniques are described in Chapter 2, Section 2.13 to 2.16. Analysis of the samples by FTIR was not conducted due to difference spectra not being able to be produced.

4.5.1 Analytical Results Obtained From Fresh Oil Samples

The analytical results obtained from the Fresh Oil samples are shown in Table 4.2

Identification Code	Oil Type	Relative Volatility	Kinematic Viscosity @ 40°C (cSt)	Kinematic Viscosity @ 100°C (cSt)	Viscosity Index	Base Number (mgKOH/g)
CAT	Mineral	1.05	111.7	13.5	118	12.3
A	Mineral	1.65	78.8	13.4	174	10.4
B	Mineral	1.60	78.4	14.1	187	10.3
C	Semi-synthetic	0.79	73.0	11.4	149	10.0
D	Synthetic	0.43	83.0	11.9	175	10.0
E	Synthetic	0.66	76.7	13.7	185	9.6
G	Mineral	0.84	108.1	13.3	120	12.5
G1	Mineral	0.95	113.7	17.1	165	8.3
H	Semi-synthetic	0.90	90.5	13.5	151	20.6
I	Synthetic	0.49	79.7	14.6	192	17.7
J	Mineral	0.56	88.2	13.9	162	16.7
K	Semi-synthetic	0.79	107.4	13.8	128	18.3
L	Synthetic	0.88	105.7	13.6	128	15.5

Table 4.2 Analytical Results from Fresh Lubricants utilised during Research Project

Examination of Table 4.2 highlights that generally mineral based lubricants are giving higher relative volatility measurements than either semi-synthetic of synthetic based lubricants. This is thought to be due to mineral based lubricants using unmodified base stocks, which will contain carbon chains of varying lengths, and hence produces higher volatility values. Semi-synthetic and synthetic based lubricants however contain carbon chains of a more 'controlled' length, which therefore produce lower volatility values. Other points to note from Table 4.2 is that oil's A to E have similar viscosity values and BN values, oil's G to I

have similar viscosity values, but differing BN values, whilst oil's J to L have similar BN values but differing viscosity values.

4.5.2 Matrix Plot and Correlation Matrix Obtained From Fresh Lubricants

The matrix plot for the Fresh lubricants is shown in Figure 4.2. The data has been coded into the oil types.

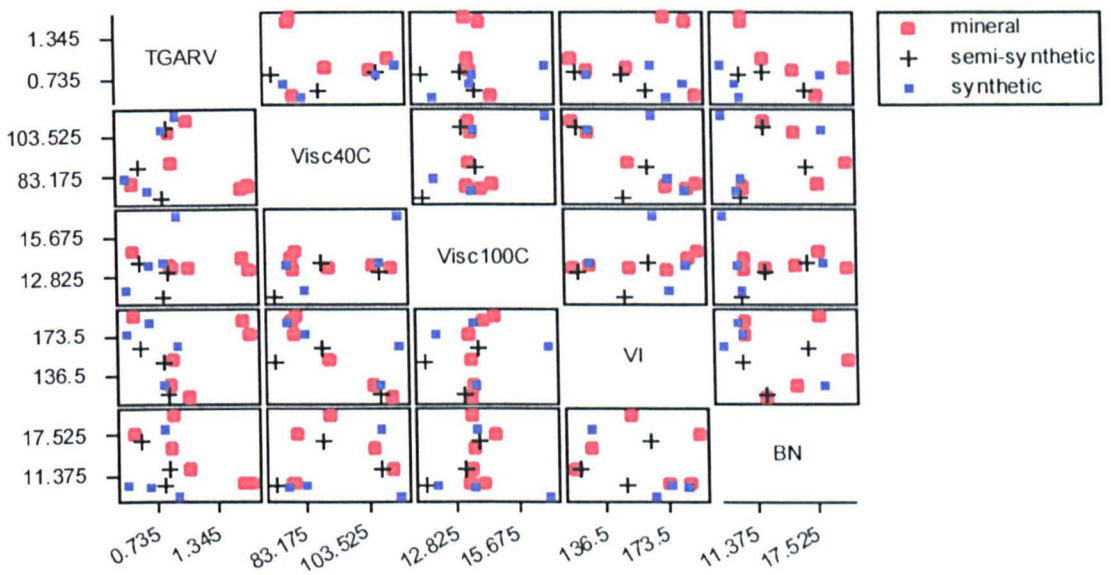


Figure 4.2 Matrix Plot for Fresh Lubricants

Examination of the Fig. 4.2 highlights that there appears to be little linear correlation with any of the measured variables, apart from Viscosity Index and Kinematic Viscosity measured at 40 °C. This is further demonstrated by the correlation matrix, shown below. (note: in this case of the correlation matrix all analytical results have been assessed, i.e. the oils have not been coded according to oil type).

Correlations (Pearson)

	TGARV	Visc40C	Visc100C	VI
Visc40C	-0.030			
	0.923			
Visc100C	0.145	0.451		
	0.637	0.122		
VI	0.033	-0.744	0.186	
	0.915	0.004	0.543	
BN	-0.300	0.160	0.003	-0.260
	0.320	0.602	0.993	0.391

Cell Contents: Correlation
P-Value

Examination of the correlation output demonstrates that only the relationship between Viscosity Index and Kinematic Viscosity at 40°C indicates a significant linear relationship. This relationship is also inverse (i.e. the higher the measured viscosity the lower the viscosity index). All the other correlation's have p-values greater than 0.05 and correlation's near to zero, thus indicating that the variables may be either acting independently of one another, or that a different correlation may exist (i.e. quadratic). Further examination of Fig. 4.2 however does not appear to highlight any polynomial relationships, therefore it appears that the relationships are independent of one another.

This lack of linear relationship may to due to the following points:-

1) A linear relationship does not exist between the measured variables measured (i.e. BN and Viscosity).
2) The lubricants used during the project came from different sources, therefore direct comparison may not be applicable (i.e. different base stocks would have been used for the mineral based lubricants).
3) The lubricants used during the project were, by design, experimental lubricants, therefore the potential linear relationship may have been lost in the formulation of these experimental lubricants.

The examination of the fresh lubricants results led to the conclusion that further analysis should not be carried out by analysing the absolute measurement. It was thought that analysing the absolute value would only highlight that the initial (i.e. fresh) measured variables were different, rather than exploring whether they were degrading differently. A key part of the project was to evaluate the performance of the lubricants, and to see how they performed under different conditions in relation to one another. Therefore the data analysis was carried out on the difference results (with respect to the fresh lubricant). The calculation used to convert all the analytical results is shown in equation 4.1

$$V_{analysis} = \frac{V_{used} - V_{fresh}}{V_{fresh}}$$ where V is the measured variable (i.e. BN)

Equation 4.1 Calculation Applied to Convert Analytical Results

It should be noted that the transformed results could be converted to percentage difference (from the fresh lubricant) by multiplying the result by 100.

4.6 Examination of the Effects of Engine Speed on the Degradation of Various Compositions of Engine Lubricant

The suite of analytical tests applied to this experiment consisted of the measurement of:-

- ZDDP P=S depletion, ZDDP POC depletion, Carbonyl oxidation formation, Nitrate Ester formation and COC oxidation formation. These chemical changes to the lubricant were measured by FTIR. The FTIR technique employed is described in Chapter 2, Section 2.13.
- Relative volatility by TGA. The TGA technique employed is described in Chapter 2, Section 2.15.
- Kinematic Viscosity at 40°C and 100°C. The Kinematic Viscosity technique employed is described in Chapter 2, Section 2.14.
- Calculation of Viscosity Index using the Viscosity at 40°C and 100°C as described in Chapter 2, Section 2.14.

The data analysis techniques employed were Principal Component Analysis (PCA), Analysis of Variance (ANOVA), matrix plots and correlation matrix.

4.6.1 Descriptive Statistics

The descriptive statistics, in relation to engine speed, sampling position, oil type and oil code are shown in Tables 4.3a to 4.3d.

MEAN VALUES

Engine Speed	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
750	0.0225168	-0.044752	-0.0264846	-0.020953	-0.322118	-0.573118	0.221691	0.63515	0.296918
1100	0.001684	-0.047667	-0.0697386	-0.06437	-0.342244	-0.456333	0.587178	0.82672	0.409789
1200	-0.0366478	0.021181	0.0654083	0.025641	-0.503833	-0.7034	0.144483	0.7651	0.623517
1400	0.0095695	-0.158314	-0.0977492	-0.150198	-0.449833	-0.475133	0.0828	0.4411	0.097733
1500	0.0382432	-0.044408	0.0021603	0.055157	-0.16545	0.03635	0.1971	0.2825	0.03885
1800	-0.0471489	0.019624	0.0578612	0.014497	-0.387167	-0.3012	0.047	1.42467	0.426717

STANDARD DEVIATION

Engine Speed	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
750	0.171923	0.102928	0.138825	0.111868	0.287042	0.302557	0.310016	0.57711	0.49743
1100	0.084746	0.108859	0.094199	0.170653	0.323397	0.432284	0.819866	0.86285	0.444849
1200	0.150433	0.13587	0.169471	0.199652	0.302955	0.301196	0.344254	0.63771	0.797306
1400	0.038491	0.139759	0.212254	0.195909	0.238467	0.423551	0.143414	0.12122	0.169279
1500	0.071226	0.01437	0.108593	0.15928	0.071913	0.316147	0.034931	0.03323	0.054942
1800	0.182799	0.117888	0.154219	0.1035	0.342862	0.349302	0.068145	1.56489	0.385378

Table 4.3a Mean Values and Standard Deviation for the Measured Variables in Relation to Engine Speed

MEAN VALUES

Position	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
P1	-0.0122991	0.016777	0.0706948	0.033176	-0.351844	-0.418294	0.18734	0.62808	0.29289
P2	0.0194891	-0.094943	-0.0802058	-0.061764	-0.272738	-0.470956	0.15589	0.46398	0.20625
P3	-0.0600649	0.028686	-0.08088	-0.151099	-0.82035	-0.6985	0.54458	2.32895	1.219
P4	-0.0095238	-0.104297	0.0074074	0.152542	-0.3752	-0.7696	1.8447	2.6653	0.7642

STANDARD DEVIATION

Position	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
P1	0.159398	0.121999	0.14315	0.135307	0.295512	0.329074	0.31832	0.81855	0.372266
P2	0.128838	0.088441	0.12027	0.152396	0.216488	0.447054	0.13136	0.20829	0.4077
P3	0.07825	0.09118	0.082828	0.077418	0.172779	0.181177	1.08915	0.67336	0.524009
P4	*	*	*	*	*	*	*	*	*

Table 4.3b Mean Values and Standard Deviation for the Measured Variables in Relation to Sampling Position

MEAN VALUES

Oil Type	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
mineral	-0.015202	-0.057332	-0.087661	-0.052874	-0.25105	-0.328875	0.506487	0.62554	0.402587
semi-synthetic	0.113924	0.028767	0.0814327	0.083893	-0.11375	-0.332067	0.139033	0.26682	0.191433
synthetic	-0.038337	-0.033034	0.0297127	-0.037776	-0.596707	-0.70248	0.038447	1.18963	0.402433

STANDARD DEVIATION

Oil Type	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
mineral	0.084603	0.079347	0.093351	0.151603	0.248334	0.340279	0.649759	0.69791	0.423762
semi-synthetic	0.022644	0.182036	0.197608	0.10711	0.111603	0.326862	0.115147	0.09142	0.252729
synthetic	0.180861	0.11783	0.134191	0.152065	0.222287	0.332471	0.12965	1.04721	0.639277

Table 4.3c Mean Values and Standard Deviation for the Measured Variables in Relation to Oil Type

MEAN VALUES

Oil Code	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
A	-0.042424	-0.072335	-0.093284	-0.058429	-0.140017	-0.290417	0.11108	0.23127	0.0358
B	-0.01875	-0.05675	-0.128133	-0.105167	-0.243817	-0.211567	0.48007	0.53695	0.466667
C	0.113924	0.028767	0.081433	0.083893	-0.11375	-0.332067	0.13903	0.26682	0.191433
CAT	0.030952	-0.035698	-0.018519	0.033898	-0.42845	-0.562525	1.13923	1.34983	0.85665
D	0.116279	-0.132129	0.121849	0.03619	-0.46155	-0.563417	0.01247	1.01472	0
E	-0.141414	0.033029	-0.031711	-0.087087	-0.686811	-0.795189	0.05577	1.30624	0.670722

STANDARD DEVIATION

Oil Code	TGARV	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE	COC
A	0.125734	0.044882	0.036787	0.035691	0.09595	0.131317	0.13133	0.14803	0.039604
B	0.050336	0.044961	0.129106	0.187793	0.165307	0.491411	0.34682	0.22916	0.426724
C	0.022644	0.182036	0.197608	0.10711	0.111603	0.326862	0.11515	0.09142	0.252729
CAT	0.029483	0.153877	0.061827	0.196079	0.425133	0.210868	1.01677	1.13371	0.198976
D	0.141077	0.1067	0.078246	0.151473	0.293758	0.293547	0.03054	1.24686	0
E	0.122155	0.070047	0.130671	0.138879	0.098501	0.339721	0.1673	0.95353	0.71606

Table 4.3d Mean Values and Standard Deviation for the Measured Variables in Relation to Oil Code

Discussion of the results in Tables 4.3a to 4.3d form part of the Principal Component Analysis section (Section 4.6.2).

4.6.2 Principal Component Analysis (PCA) of Dataset

The PCA was conducted in SCAN using the correlation matrix approach. The correlation matrix standardises the variables, thus giving them the same scaled units. This removes the effects differently scaled variables may by giving them the same weighting. Therefore the PCA is analysing the relative effects of the variables, rather than highlighting that they have

different orders of magnitude. The tabulated PCA analysis of the Correlation Matrix is shown below.

Principal Component Analysis
Calculated from Correlation Matrix by SVD

Eigenvalue	2.769	1.894	1.322	0.962	0.738	0.558
Proportion	0.308	0.210	0.147	0.107	0.082	0.062
Cumulative	0.308	0.518	0.665	0.772	0.854	0.916
Eigenvalue	0.460	0.211	0.086			
Proportion	0.051	0.023	0.010			
Cumulative	0.967	0.990	1.000			
Eigenvectors						
Variable	PC1	PC2	PC3	PC4	PC5	PC6
TGARV	0.255	-0.159	-0.448	-0.204	0.781	0.037
Visc40C	-0.099	-0.258	0.673	0.128	0.439	-0.283
Visc100C	-0.003	-0.698	0.033	-0.008	-0.056	-0.220
VI	0.040	-0.625	-0.253	0.194	-0.324	0.217
ZDDP POC	0.518	-0.041	0.096	0.273	0.085	0.340
ZDDP P=S	0.420	0.119	0.176	0.493	-0.061	-0.266
C Ox	-0.223	0.112	-0.410	0.719	0.155	-0.193
NE	-0.499	-0.034	-0.145	0.039	0.066	-0.299
COC	-0.425	-0.039	0.226	0.263	0.221	0.714
Variable	PC7	PC8	PC9			
TGARV	0.146	0.132	-0.129			
Visc40C	-0.265	-0.152	-0.296			
Visc100C	0.063	0.216	0.640			
VI	-0.020	-0.084	-0.589			
ZDDP POC	0.044	-0.658	0.298			
ZDDP P=S	0.545	0.377	-0.148			
C Ox	-0.417	0.010	0.136			
NE	0.612	-0.509	-0.017			
COC	0.239	0.268	0.098			

Examination of the PCA output highlights that the first two Principal Components (PC) account for 52% of the variation in the data. This is considered to be less than ideal, therefore interpretation of the results have to be treated with caution, due to 48% of the variation the data not being accounted for by the 1st two PC's.

The Principal Component Loading Plot for PC1 verses PC2 is shown in Figure 4.3.

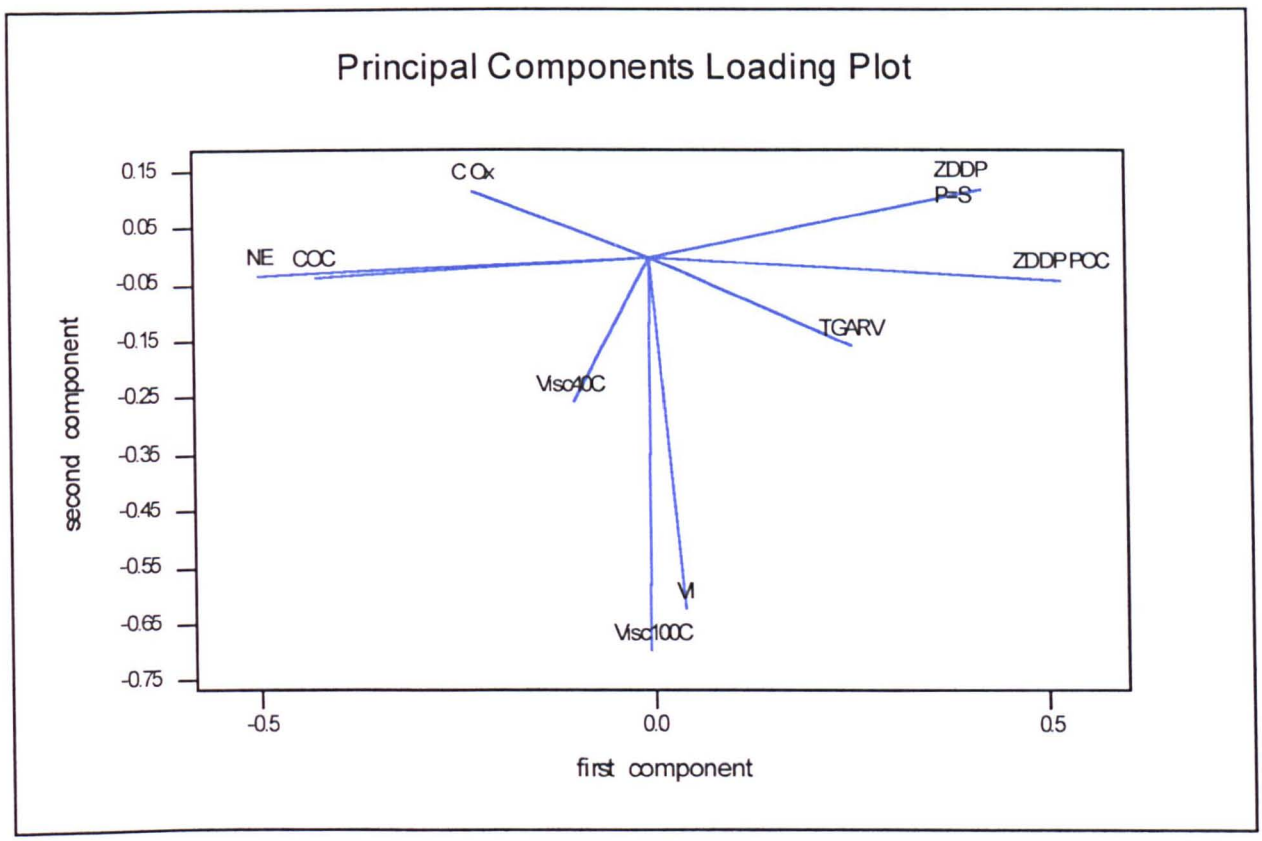


Fig. 4.3 Principal Component Loading Plot for PC1 verses PC2

Examination of the PC Loading Plots highlights that:-

PC1 is influenced by,

- ZDDP POC, ZDDP P=S and TGARV in a positive direction, although ZDDP POC has the most influence.
- NE, COC, C Ox in a negative direction, although NE and COC have more of an effect.
- Visc40C, Visc100C, VI have little influence on PC1.

PC2 is influenced by,

- Visc100C, VI, Visc40C and TGARV in a negative direction, although Visc100C and VI have a far greater influence.
- C Ox and ZDDP P=S in a positive direction, although the effect is small.
- NE, COC, and ZDDP POC have little influence on PC2.

Interpretation of the PC loading plots is not an easy task, although it appears that PC1 is more influenced by chemical changes to the lubricant, which is demonstrated by the inclusion of the ZDDP depletion and the oxidant formation. PC2 appears to be more influenced by physical changes, which is demonstrated by the inclusion of the changes in viscosity.

The Principal Component Score plot with respect to engine speed on the measured variables is shown in Figures 4.4.

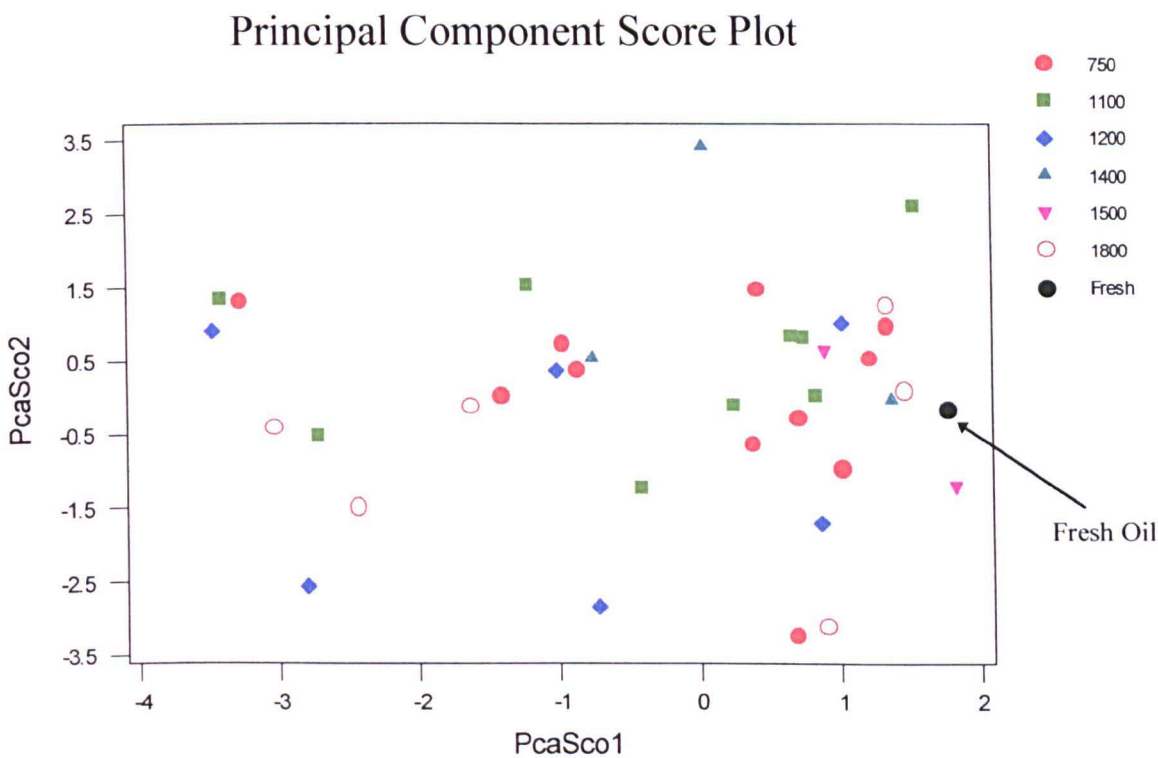


Fig. 4.4 Principal Component Score Plot for PC1 and PC2 (with respect to engine speed in rpm)

Examination of Fig. 4.4 highlights that there is a separation in the group of points on PC1 into three groups, as the majority of points lie on the positive side of PC1, whilst a smaller two groups of points lie on the negative side of PC1. Reviewing Fig. 4.4 against Fig. 4.3 highlights that the group towards the positive side of PC1 is more influenced by their ZDDP POC and ZDDP P=S levels. The groups that lies to on the negative side of PC1 are more influenced by their NE and COC levels.

Examination of PC1

Close examination of the data highlights that the further the data point is to the left from the Fresh oil point the more chemically degraded the lubricant is, in that the Nitrate Ester levels are high, the COC Oxidation levels are high and the ZDDP POC and ZDDP P=S depletion is greatest. Therefore the point furthest to the left of the fresh oil point is the most chemically degraded.

Examination of PC2

Close examination of the data highlights that points above the Fresh oil point have a viscosity less than the fresh oil sample, whilst points below the Fresh oil point have viscosity levels greater than the fresh oil sample. The reason for changes in viscosity have already been discussed in Chapter 2, Section 2.14.3, however in summary the reduction in viscosity is either due to fuel dilution, shearing of long chain molecules or water contamination, whilst an increase in viscosity is either due to loss of the volatile light ends of the lubricant, by the loading of the lubricant with insoluble matter or oxidation of the base oil. Changes in viscosity are undesirable in lubricants. If the viscosity reduced is too much the outcome will be reduction in the film thickness of the lubricant on metallic parts, which will lead to mechanical wear. If the viscosity increases too much the flow properties of the lubricant will be effected. Therefore upon cold start ups the lubricant will not be able to coat the key metallic parts in the upper part of the engine, which will lead to mechanical wear. It is not possible to definitively say that the viscosity changes noted in PC2 are sufficient to give rise to the scenario's above, however if the increase or decrease viscosity trend continued then engine wear problems could result.

Further examination of Figures 4.4 highlights that different engine speeds do not appear to show any discernible pattern in the data. No particular engine speed appears to stand out. This highlights that engine speed may not be having an effect on lubricant degradation. It would be assumed that increased engine speeds would degrade the lubricants more due to increased engine temperatures and increased internal motion of the piston in the cylinder bore. The reasons why the engine speed appears to have had no influence on the lubricant degradation remains unclear, however it may be due to the fact that the changes in engine

speed did not increase the engine temperature sufficiently to produce an increased level of lubricant degradation.

The Principal Component Score plots with respect to sampling position on the measured variables is shown in Figures 4.5.

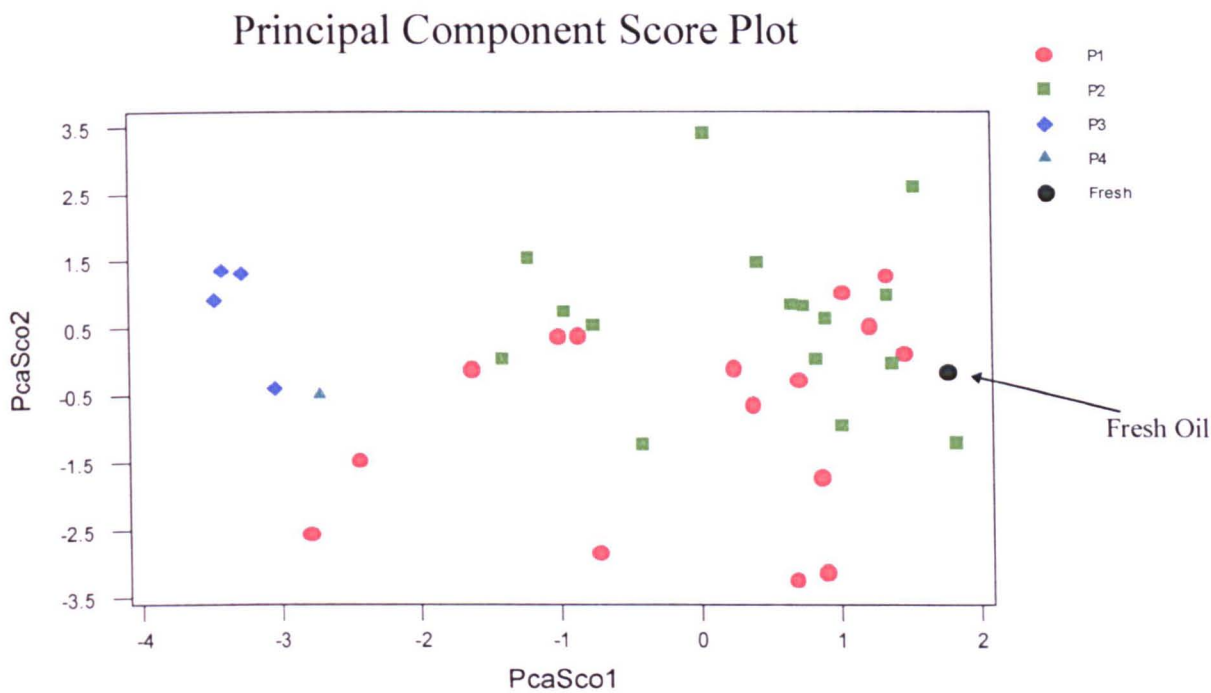


Fig. 4.5 Principal Component Score Plot for PC1 and PC2 (with respect to sampling position)

Examination of Fig. 4.5 highlights that the group on the furthest negative side of PC1 contains samples predominately taken from sampling positions nearer the combustion chamber (position 3 was taken from behind the 1st compression ring, whilst position 4 was taken from the base of the crown land). It can therefore be concluded that samples from nearer the combustion chamber contain greater levels of nitrate ester, COC oxidation and having their ZDDP reserves depleted (i.e. the lubricant samples are more chemically degraded). This is most probably due to the oil samples being subjected to increased temperatures by their proximity to the combustion chamber, and therefore more chance of being subjected to combustion gases.

The above results are not too surprising, because it highlights that the oil samples from positions 1 and 2 are less chemically degraded that oil samples taken from position 3 and 4.

Examination of Fig. 4.5 in terms of PC2 highlights that there are more points from sampling Position 2 above the fresh oil point, whilst there are more points from sampling position 1 below the fresh oil point. Reviewing the previous findings highlights that samples produced from sample position 2 have lower viscosity values, when compared to the fresh oil samples, whilst samples produced from sample position 1 have higher viscosity values, when compared to fresh oil samples. Review of Table 4.3b also highlights this trend. These differences highlight that the lubricant behaves differently when sampled from behind a piston ring (position 1) compared to the piston face (position 2). The samples taken from position 3 appear to be behaving more like samples from position 2.

The Principal Component Score plot with respect to oil type on the measured variables is shown in Figures 4.6.

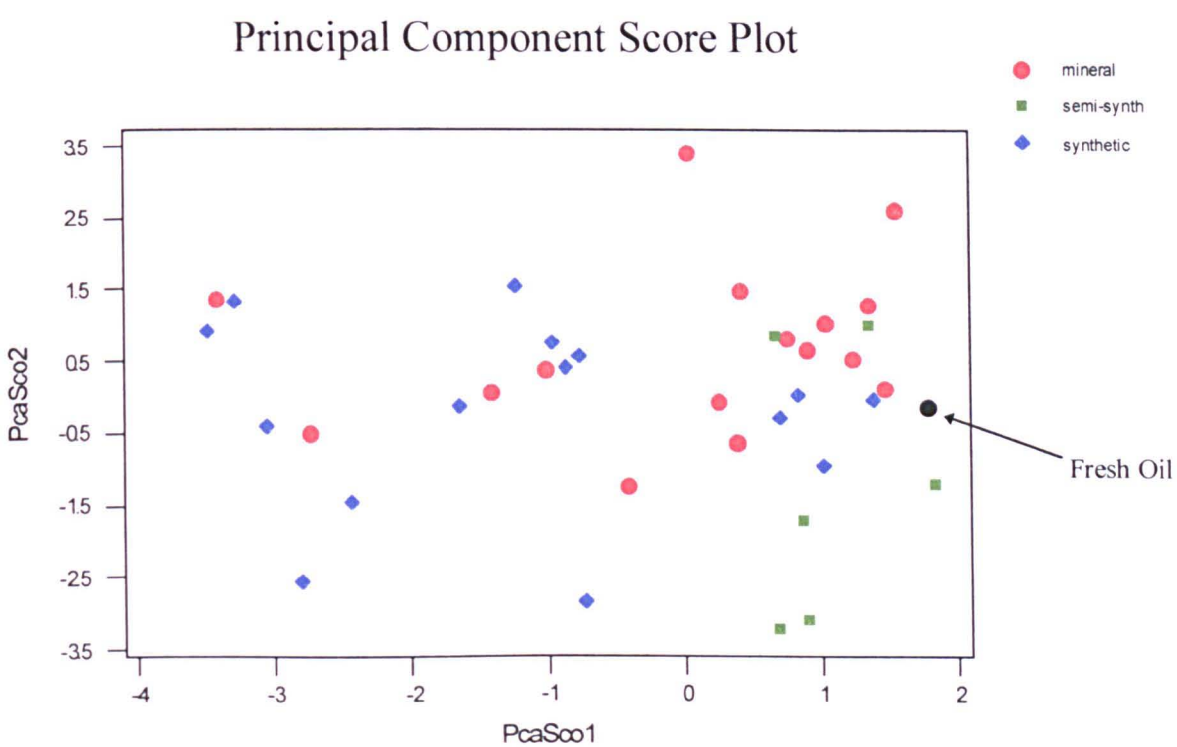


Fig. 4.6 Principal Component Score Plot for PC1 and PC2 (with respect to oil type)

Examination of Fig. 4.6 highlights that mineral based lubricants are producing more points above the fresh oil point (i.e the viscosity has decreased when compared to the fresh lubricant). Assuming that the chance of fuel and water contamination of the oil sample was equal for whatever oil type was used, this leads to the conclusion that the mineral based lubricants were shearing more that the synthetic based lubricants. The semi-synthetic lubricants are generally below the fresh oil point (i.e the viscosity has increased when compared to the fresh lubricant). This indicates that the semi-synthetic lubricant is either losing 'light ends' of the lubricant, being more prone to insoluble loading (i.e. dispersant additives are not performing adequately), or the base oil has become oxidised. Referring to Table 4.3c demonstrates that the mean oxidation levels, measured as Carbonyl oxidation, Nitrate Ester Formation and COC oxidation, for the semi-synthetic lubricant is less than the mineral based lubricant, and in the case of Nitrate Ester and COC oxidation, less than the synthetic lubricant. Therefore it seems unlikely that oxidation of the semi-synthetic base oil has caused the viscosity increase. Also Table 4.3c demonstrates that the mean relative volatility change for the semi-synthetic lubricant has shown an increase, which should show a corresponding decrease in viscosity. Therefore the increase in viscosity seen in the semi-synthetic lubricant is pointing towards the loading of insoluble matter in the lubricant, which possibly highlights the dispersant additives are not performing adequately. It should be noted that no discernible pattern can be noted with the synthetic lubricant (i.e. oils have shown no viscosity change, either a viscosity increase or a viscosity decrease).

Further examination of Fig 4.6 does not suggest that any particular type of lubricant was fairing any better than any other, due to the scatter in the data (note: the points on PC1, which indicate level of chemical degradation, contain both mineral and synthetic lubricants). However it should be pointed out that it proved impossible to obtain oil samples at sampling positions 3 and 4 for many of the mineral based lubricants, where an oily carbon containing soot was obtained, suggesting that these lubricants were not fairing well under these conditions. The indications are that had a sufficient oil sample been obtained, then these lubricants would have shown a larger level of degradation than the synthetic lubricants.

The Principal Component Score plot with respect to oil code on the measured variables is shown in Figures 4.7.

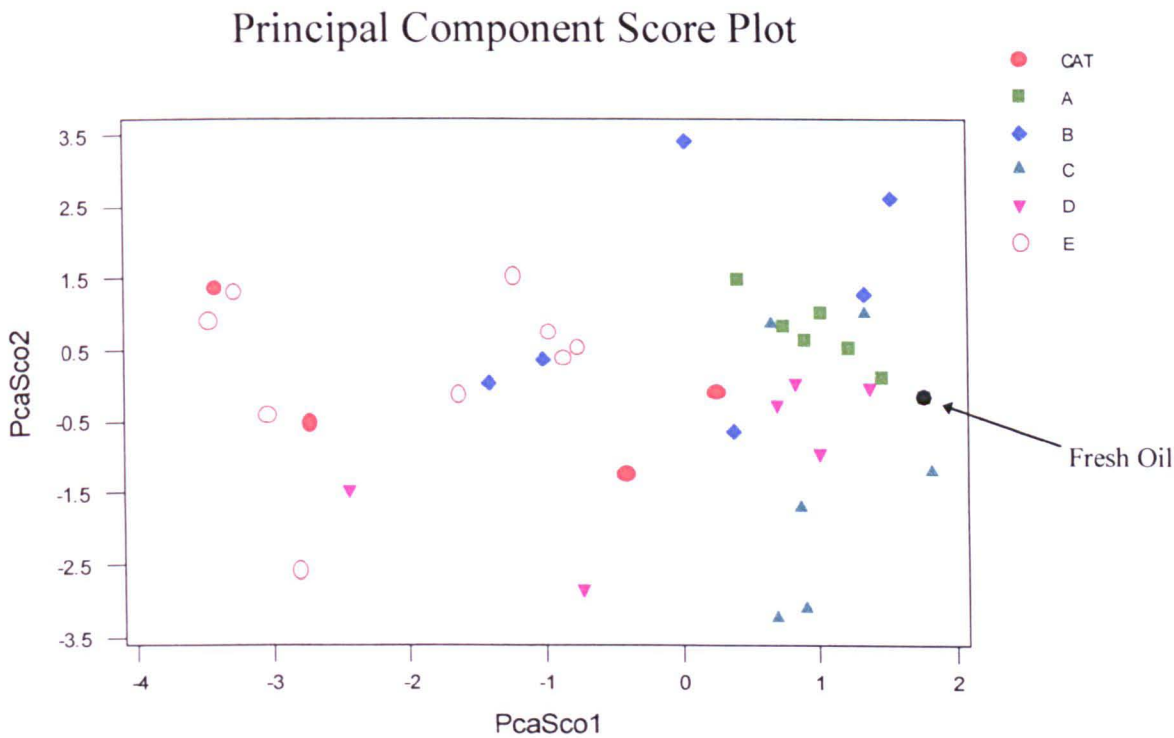


Fig. 4.7 Principal Component Score Plot for PC1 and PC2 (with respect to oil code)

Examination of Fig. 4.7 highlights the point made previously that no particular lubricant stands out as being better than the rest, i.e. being very close to the fresh oil point. An argument could be made that would suggest that the 'best' lubricant is oil A, however this oil could not provide a liquid oil sample at Position 3. This was thought to be due to the poor volatility characteristic of this particular lubricant, ref. Table 4.2. Taking this information into account it would be suggested that oil A is the 'worst' oil, whilst oils D and E are 'better' oils, due to them being able to produce liquid oil samples at position 3.

4.6.3 Possible Screening Test for Lubricant Quality Using PCA

Using the knowledge that PC1 and PC2 are representing changes it is possible to code the PC output, according to either the level of viscosity change or the level of chemical degradation. By simply summing the chemical degradation (i.e. adding the change noted in Carbonyl oxidation, Nitrate Ester formation, COC oxidation and the depletion in ZDDP

P=S and ZDDP POC) or the viscosity change, a single numerical value associated with the level of chemical degradation or viscosity change is arrived at. If a code is then applied to the level of the numerical value, the plots shown in Fig. 4.8 and 4.9 are produced. In terms of chemical degradation the points have been coded as fresh, low, medium and high, whilst the viscosity change has been coded as fresh, same, increase or decrease.

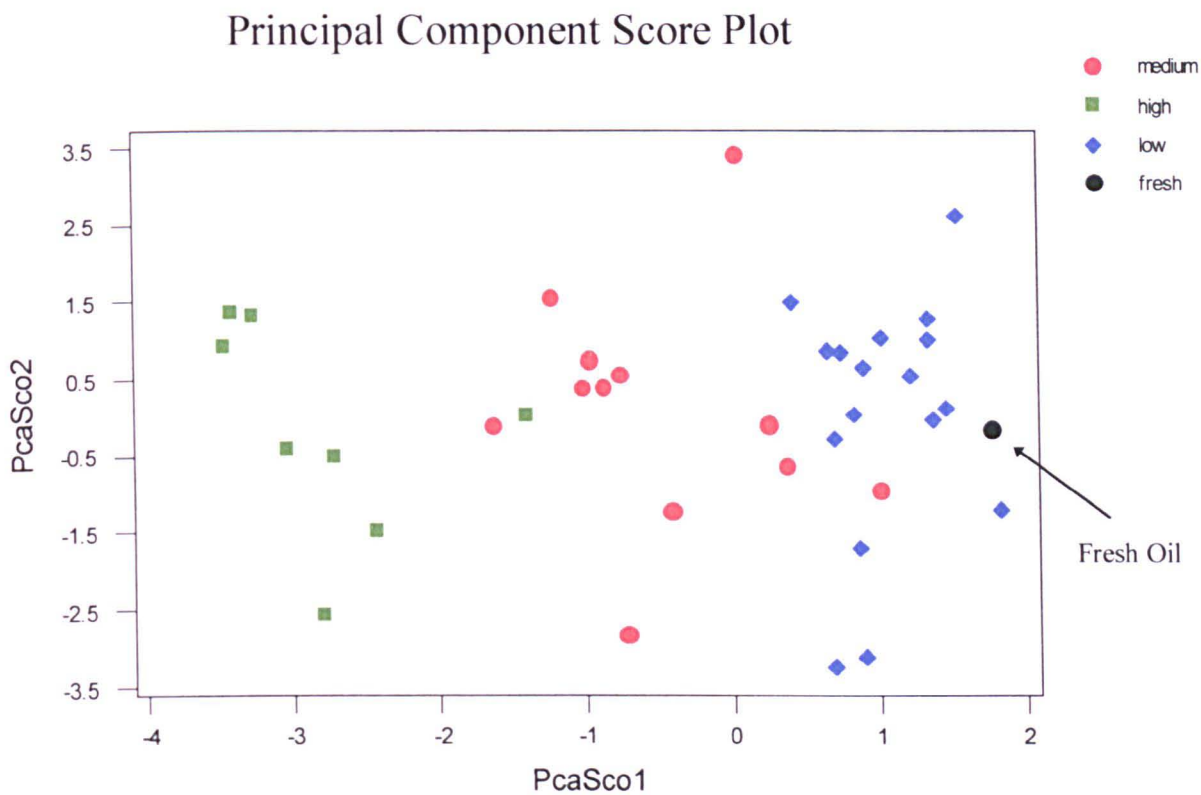


Fig. 4.8 Principal Component Score Plot for PC1 and PC2 (highlighting level of chemical degradation of the lubricants)

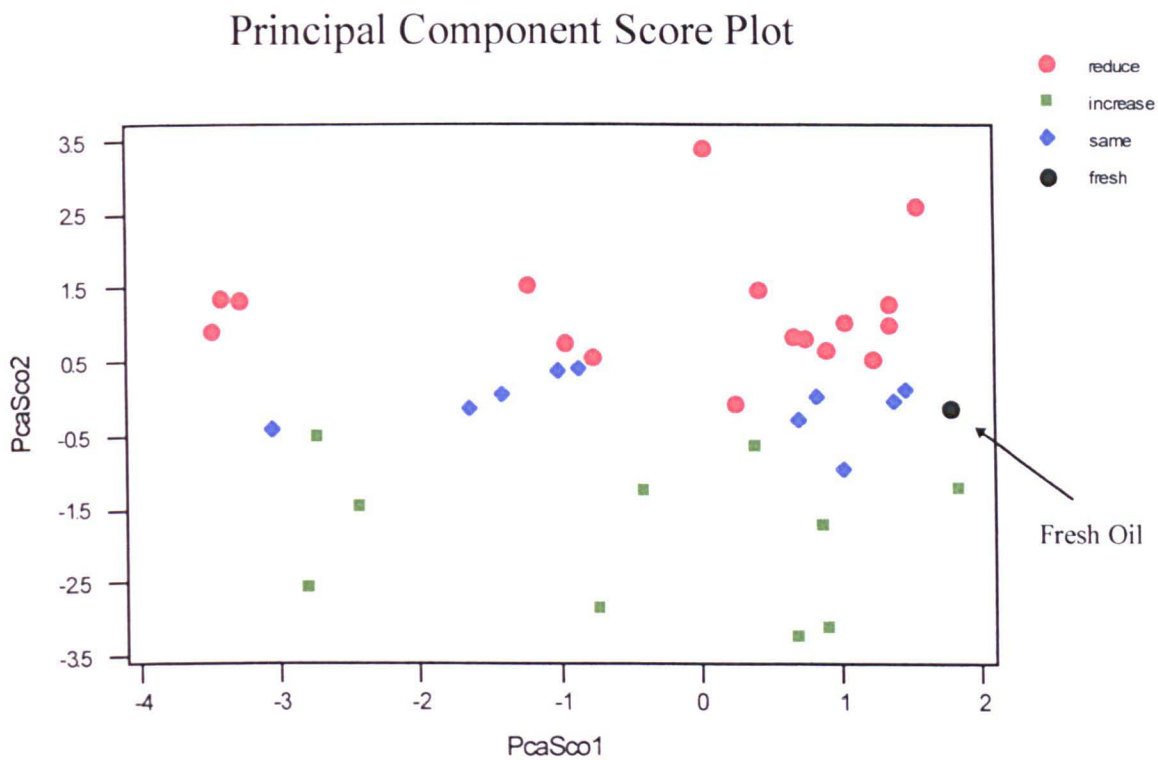


Fig. 4.9 Principal Component Score Plot for PC1 and PC2 (highlighting viscosity change of the lubricants)

Examination of Fig. 4.8 and 4.9 highlights that the arbitrary assigning of codes has generally coded the level of chemical degradation and viscosity change correctly. It should be noted that the coding of the levels has been chosen arbitrarily, therefore the assigned code may not be considered to be actually 'high' or 'low' when compared to lubricant degradation in standard engine tests.

It is therefore thought that this approach could be used as a potential quick screening of lubricant performance before an expensive full scale lubricant trial was initiated. An example on how to do this would be to 'bench mark' the ring zone sampling on an engine (note: the CAT 3406B may not be the ideal engine for this type of work). This could be done by running standard lubricants (i.e. the sequence III lubricants, or lubricants which have historically performed 'well' and 'badly' in the field) and taking samples from the ring zone. These ring zone samples would then subjected to a pre-defined suite of analytical tests (e.g. Viscosity, BN, FTIR, volatility, ICP-AES) which would allow the generation of information

on the chemical and physical degradation of the lubricant, as well as information on engine wear. The analytical results could then coded in a similar fashion (i.e. summing the chemical degradation, viscosity change, engine wear etc.). These analytical results could then be analysed by PCA, and the resulting PCA plots would be plotted against the assigned codes. Assuming similar plots to Fig. 4.8 and 4.9 are produced a multivariate quality control chart will have been produced. Test lubricants could then be run and ring zone samples taken. If these samples were then subjected to the same suite of analytical tests the results could then be related to the multivariate quality control chart. Ideally this would then highlight quickly whether the lubricant was 'good', 'average' or 'bad'. This would then provided information on whether more extensive engine tests should be carried out on the lubricant. The advantage to using the above described approach is that:-

- ring zone sampling is quicker than sump sampling (i.e. similar degradation will be seen in ring zone samples after a few hours, compared to 100+ hours needed for sump sampling).
- The use of PCA reduces the number a dimensions in a data set to a more manageable number, ideally into one bivariate plot. This makes interpretation of the data, and hence conclusions and recommendations, much simpler

4.6.4 ANOVA Analysis of Dataset

Analysis of Variance was carried out to examine the findings noted from the Principal Component Analysis. The ANOVA output is shown below.

General Linear Model

```
Factor      Type Levels Values
Type       fixed      3 mineral semi-synthetic synthetic
Speed      fixed      6  750 1100 1200 1400 1500 1800
Position   fixed      4 P1 P2 P3 P4

Analysis of Variance for TGARV, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Type         2     1031.2      940.7      470.3    2.28  0.123
Speed        5      290.0      187.1       37.4    0.18  0.967
Position     3       14.4       14.4        4.8    0.02  0.995
Error       26     5374.3     5374.3      206.7
Total       36     6709.9
```

Analysis of Variance for Visc40C, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	323.6	312.3	156.1	1.31	0.288	
Speed	5	773.0	202.4	40.5	0.34	0.885	
Position	3	667.0	667.0	222.3	1.86	0.161	
Error	26	3105.0	3105.0	119.4			
Total	36	4868.6					

Analysis of Variance for Visc100C, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	1700.5	1900.6	950.3	7.47	0.003	SIG
Speed	5	838.7	250.0	50.0	0.39	0.849	
Position	3	1634.1	1634.1	544.7	4.28	0.014	SIG
Error	26	3307.8	3307.8	127.2			
Total	36	7481.2					

Analysis of Variance for VI, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	860.2	603.5	301.7	1.54	0.233	
Speed	5	700.5	454.0	90.8	0.46	0.800	
Position	3	1465.3	1465.3	488.4	2.49	0.082	
Error	26	5093.0	5093.0	195.9			
Total	36	8119.0					

Analysis of Variance for ZDDP POC, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	1.39127	0.89248	0.44624	11.24	0.000	SIG
Speed	5	0.11761	0.11247	0.02249	0.57	0.725	
Position	3	0.52900	0.52900	0.17633	4.44	0.012	SIG
Error	26	1.03248	1.03248	0.03971			
Total	36	3.07035					

Analysis of Variance for ZDDP P=S, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	1.23916	0.78693	0.39346	4.03	0.030	SIG
Speed	5	0.87551	1.00763	0.20153	2.07	0.102	
Position	3	0.40644	0.40644	0.13548	1.39	0.268	
Error	26	2.53662	2.53662	0.09756			
Total	36	5.05772					

Analysis of Variance for C Ox, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	1.7959	0.9310	0.4655	3.69	0.039	SIG
Speed	5	0.7641	0.6273	0.1255	1.00	0.440	
Position	3	2.5938	2.5938	0.8646	6.86	0.001	SIG
Error	26	3.2765	3.2765	0.1260			
Total	36	8.4303					

Analysis of Variance for NE, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	4.4693	1.9601	0.9801	3.36	0.050	SIG
Speed	5	3.5294	2.1742	0.4348	1.49	0.227	
Position	3	11.5902	11.5902	3.8634	13.25	0.000	SIG
Error	26	7.5814	7.5814	0.2916			
Total	36	27.1703					

Analysis of Variance for COC, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Type	2	0.2240	0.1524	0.0762	0.41	0.667	
Speed	5	0.8858	0.4752	0.0950	0.51	0.764	
Position	3	3.0366	3.0366	1.0122	5.47	0.005	SIG
Error	26	4.8120	4.8120	0.1851			
Total	36	8.9584					

The ANOVA analysis demonstrates that the significant effects (i.e. p-value less than 0.05) on the measured variables are:-

- TGA has no significant effects, although oil type is only marginally non-significant.
- Kinematic Viscosity at 40°C has no significant effects, although sampling position is only marginally non-significant.
- Kinematic Viscosity at 100°C is significantly effected by Oil Type and Sampling Position.
- Viscosity Index has no significant effects, although sampling position is only marginally non-significant.
- ZDDP POC is significantly effected by Oil type and Sampling Position.
- ZDDP P=S is significantly effected by Oil Type, although engine speed is only marginally non-significant.
- Carbonyl Oxidation is significantly effected by Oil Type and Sampling Position.
- Nitrate Ester is significantly effected by Oil Type and Sampling Position.
- COC Oxidation is significantly effected by Sampling Position.

These findings are demonstrated graphically in the main effects plots, Fig 4.10a to 4.10i.

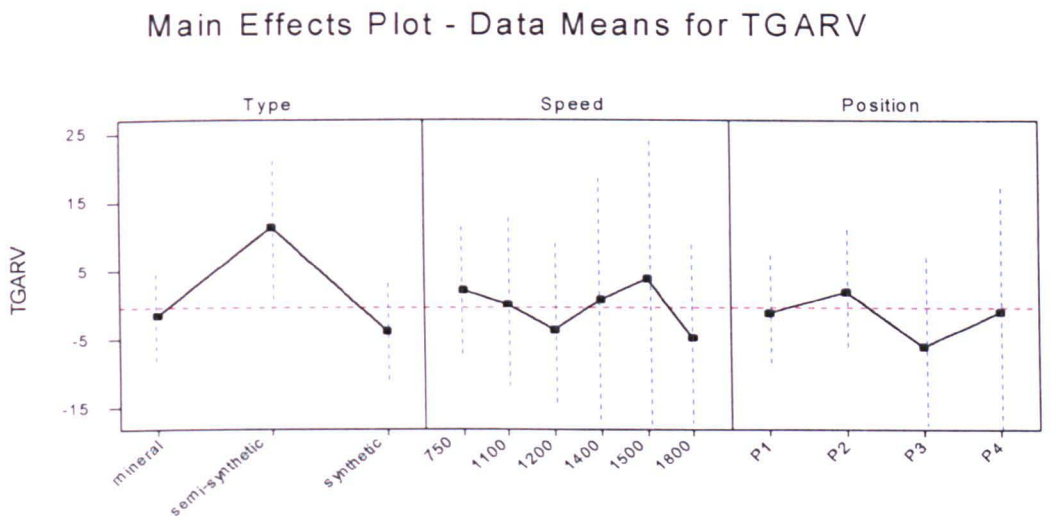


Fig. 4.10a Main Effects Plot Highlighting the Percentage Change in Relative Volatility with Respect to Oil Type, Engine Speed and Sampling Position.

Fig. 4.10a demonstrates that there are no significant effects (although oil type is only marginally non-significant).

Fig. 4.10b Main Effects Plot Highlighting the Percentage Change in Kinematic Viscosity at 40°C with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10b demonstrates that there are no significant effects (although sampling position is only marginally non-significant).

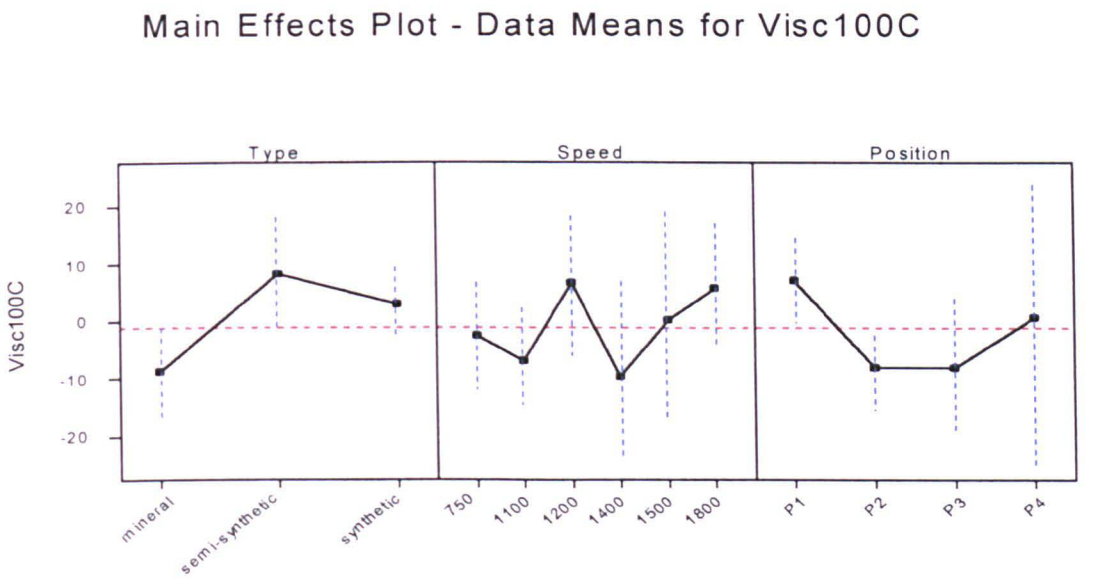


Fig. 4.10c Main Effects Plot Highlighting the Percentage Change in Kinematic Viscosity at 100°C with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10c demonstrates that the significant effects are oil type and sampling position.

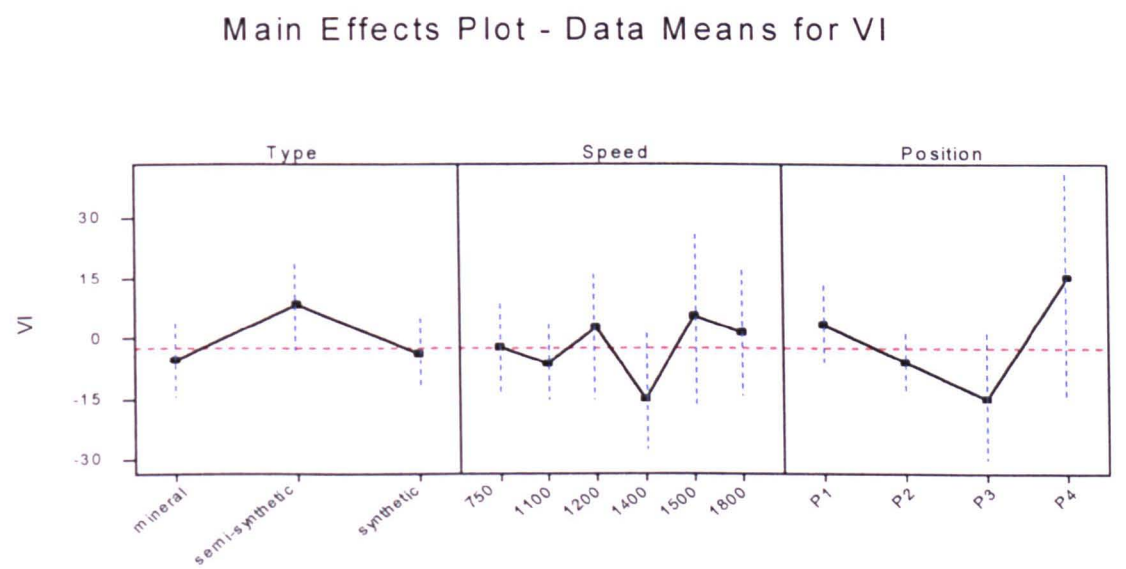


Fig. 4.10d Main Effects Plot Highlighting the Percentage Change in Viscosity Index with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10d demonstrates that there are no significant effects (although sampling position is only marginally non-significant).

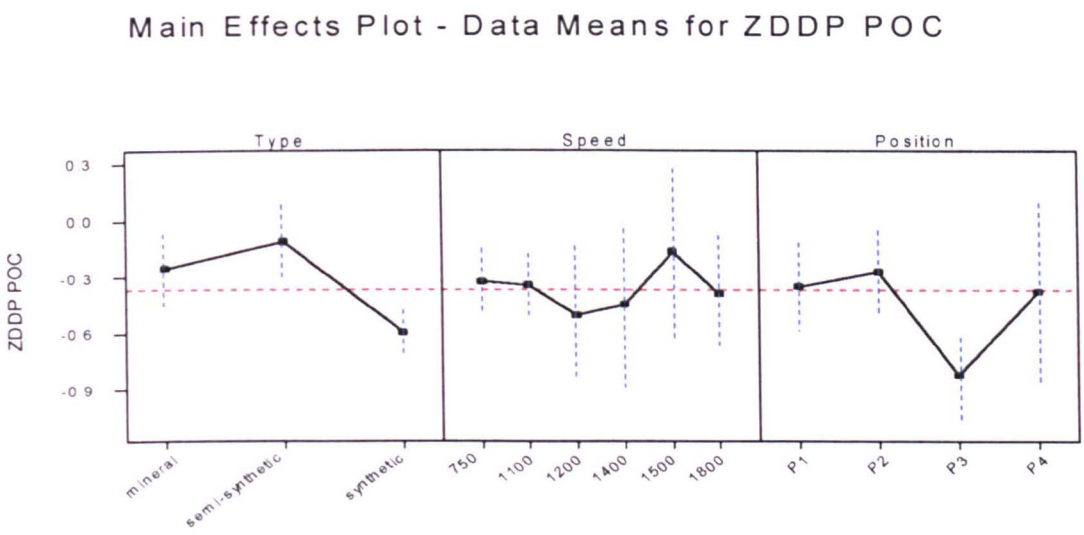


Fig. 4.10e Main Effects Plot Highlighting the Loss of ZDDP POC with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10c demonstrates that the significant effects are oil type and sampling position.

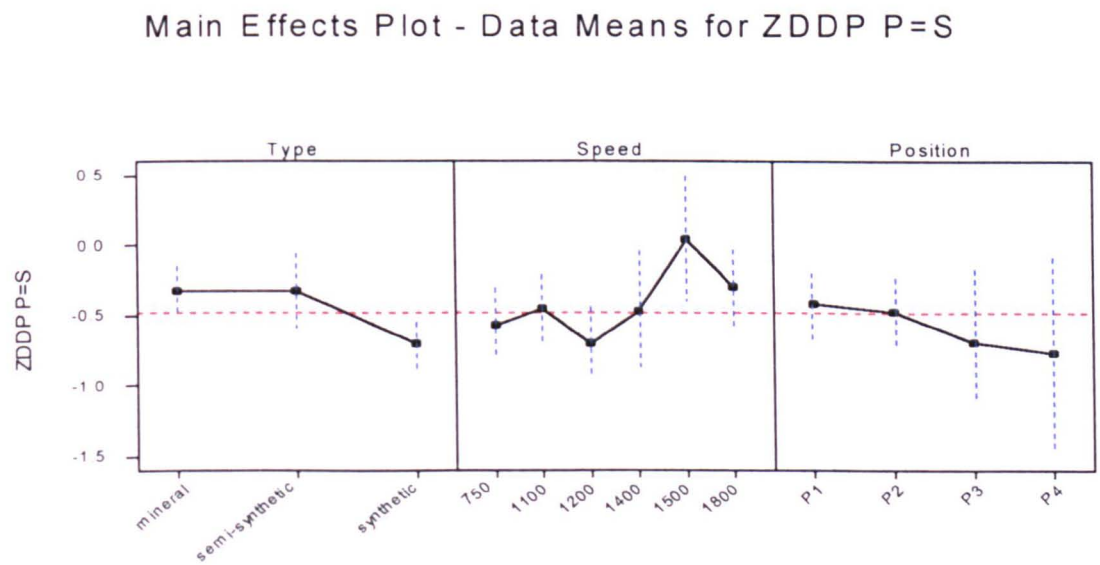


Fig. 4.10f Main Effects Plot Highlighting the Loss of ZDDP P=S with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10f demonstrates that the significant effects is oil type (although engine speed is only marginally non-significant).

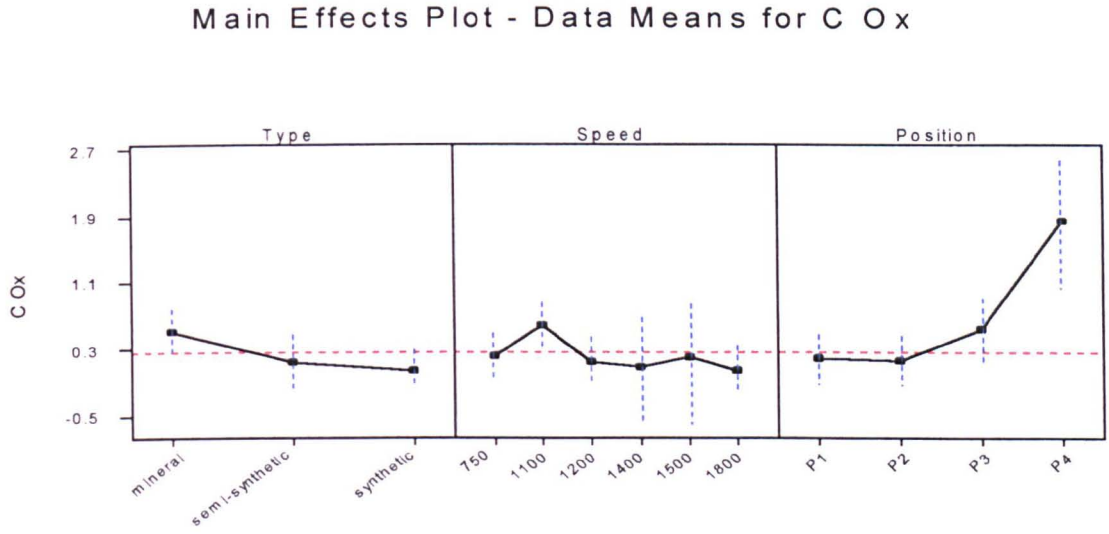


Fig. 4.10g Main Effects Plot Highlighting the Increase in Carbonyl Oxidation with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10g demonstrates that the significant effects are oil type and sampling position.

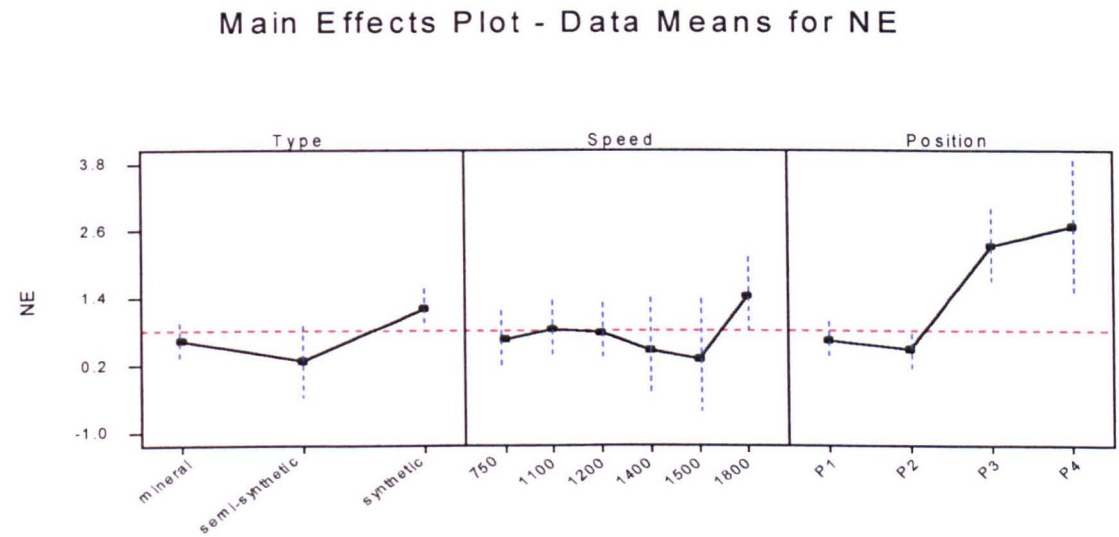


Fig. 4.10h Main Effects Plot Highlighting the Increase in Nitrate Ester with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10h demonstrates that the significant effects are oil type and sampling position.

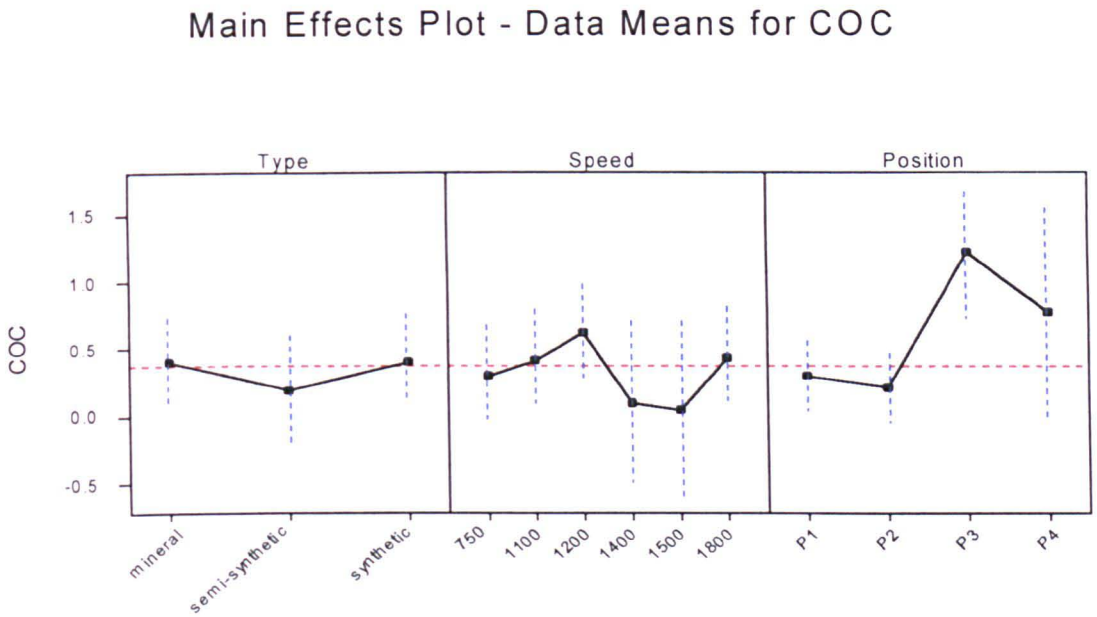


Fig. 4.10i Main Effects Plot Highlighting the Increase in COC Oxidation with Respect to Oil Type, Engine Speed and Sampling Position

Fig. 4.10i demonstrates that the significant effect is sampling position.

4.6.5 Correlation's within the Dataset

The correlation's with the measured variables are examined by the matrix plot, Fig. 4.11.

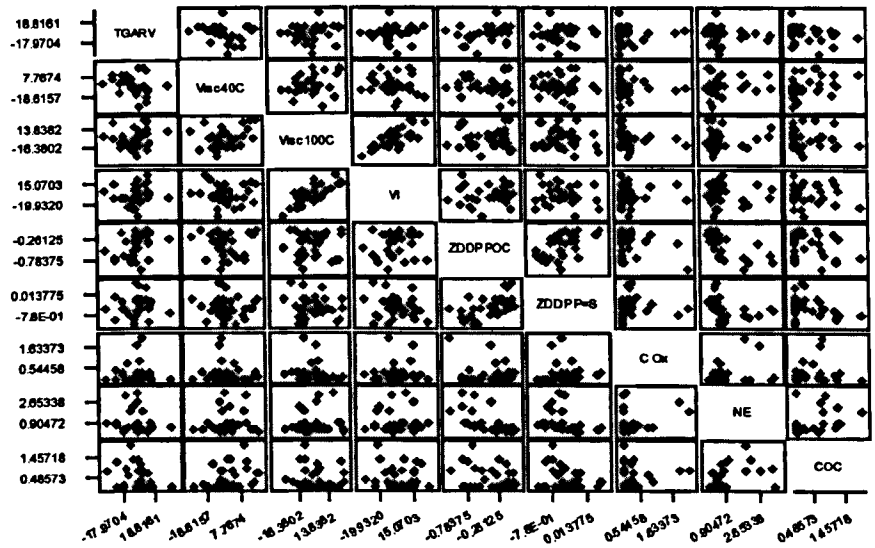


Fig. 4.11 Matrix Plot of Measured Variables

Examination of the matrix plot demonstrates that there are potential linear trends according with the degradation of the lubricants. This is further demonstrated by the correlation matrix, shown below.

Correlations (Pearson)

	Visc40C	Visc100C	VI	ZDDP POC	ZDDP P=S	C Ox	NE
Visc100C	0.354						
	0.031						
VI	-0.034	0.762					
	0.844	0.000					
ZDDP POC	-0.076	-0.023	0.128				
	0.655	0.894	0.452				
ZDDP P=S	-0.063	-0.125	-0.123	0.563			
	0.713	0.462	0.470	0.000			
C Ox	-0.124	-0.153	0.064	-0.158	0.016		
	0.463	0.367	0.705	0.351	0.927		
NE	0.081	0.087	0.014	-0.645	-0.359	0.283	
	0.633	0.610	0.932	0.000	0.029	0.090	
COC	0.331	0.000	0.012	-0.315	-0.281	0.174	0.437
	0.045	0.999	0.946	0.058	0.092	0.302	0.007

Cell Contents: Correlation
P-Value

The correlation's that are significant (i.e. p-values less than 0.05) are between Viscosity at 40°C and Viscosity at 100°C, Viscosity at 40°C and COC oxidation, Viscosity at 100°C and Viscosity Index, ZDDP POC depletion and ZDDP P=S depletion, ZDDP P=S depletion and Nitrate Ester formation, Nitrate Ester formation and COC oxidation. There are some marginally non-significant correlation's also between ZDDP POC depletion and COC oxidation, ZDDP P=S depletion and COC oxidation and Carbonyl oxidation and Nitrate Ester formation.

The number of significant, and marginally significant correlation's demonstrate the complex nature of lubricants and how they degrade. This further highlights the need to use multivariate analysis to analyse the data, and to gain the fullest understanding. An interesting point to note is that there appeared to be little correlation with the fresh lubricants (ref: Cheaper 4, Section 4.5.2), however correlation's do exist with the used lubricants. This demonstrates that some of the measured variables do change/degrade in a linear manner.

4.6.6 Conclusions from PCA and ANOVA

The conclusions from the PCA and ANOVA analysis are:-

1. The PCA analysis showed that there is a relationship between the chemical degradation, which is highlighted by how far left the points are on PC1. PC2 highlighted that points above the fresh oil demonstrated a viscosity decrease, while points below the fresh oil demonstrated a viscosity increase.
2. Engine speed has no effect on lubricant degradation.
3. Samples from position 3 are more chemically degraded that samples from position 1 and position 2. This is due to the samples from position 3 being taken nearer the combustion chamber.
4. Samples from position 2 generally have viscosity values lower than the fresh oil samples. Samples from position 1 generally have viscosity values higher than the fresh oil samples.
5. Mineral based lubricants generally show a decrease in viscosity, which is thought to be due to shearing of long chain molecules. Semi-synthetic based lubricants show a viscosity increase, which is though to be due to the level of insoluble matter.

6. No particular lubricant stands out as being 'better' than another, although only oil D and E produced oil samples from position 3.
7. PCA analysis could be used along with ring zone sampling to produce a quick screening test for lubricant quality.
8. ANOVA analysis showed that there were significant linear relationships for Kinematic viscosity at 100°C (Oil Type and Sampling Position), ZDDP POC (Oil type and Sampling Position), ZDDP P=S (Oil Type), Carbonyl Oxidation (Oil Type and Sampling Position), Nitrate Ester (Oil Type and Sampling Position), and COC Oxidation (Sampling Position).
9. The variables showed linear correlation's between Viscosity at 40°C and Viscosity at 100°C, Viscosity at 40°C and COC oxidation, Viscosity at 100°C and Viscosity Index, ZDDP POC depletion and ZDDP P=S depletion, ZDDP P=S depletion and Nitrate Ester formation, Nitrate Ester formation and COC oxidation.

4.7 Examination of the Effects of Fuel Sulphur Content on the Degradation of Various Compositions of Engine Lubricant.

Due to the results from the previous experiment examining the effects of engine speed and sampling position on lubricant degradation, where it was found that engine speed had little effect on lubricant degradation, it was decided that the engine would be run at a constant speed. A engine speed of 1300rpm was chosen because it represented a 'medium' running speed and this engine speed prolonged the life of the sampling system.

Also lubricant samples were only taken from the sump and from behind the two compression rings (Position 1 and Position 3). This decision was made because the lack of lubricant being obtained from Position 4 was making analysis very difficult and that engine strip downs had to take place every time samples were needed to be taken from Position 2 and Position 4 (ref: Chapter 2, Section 2.4).

The suite of analytical tests applied to this experiment consisted of the measurement of:-

- Relative volatility by TGA. The TGA technique employed is described in Chapter 2, Section 2.15.

- Kinematic Viscosity at 40°C and 100°C. The Kinematic Viscosity technique employed is described in Chapter 2, Section 2.14.
- Calculation of Viscosity Index using the Viscosity at 40°C and 100°C as described in Chapter 2, Section 2.14.
- Base Number. The Base Number technique employed is described in Chapter 2, Section 2.16.
- Particle Size Analysis by Laser Light Diffraction. The Particle Size Analysis technique is described in Chapter 2, Section 2.17.

The data analysis techniques employed were Principal Component Analysis (PCA), Analysis of Variance (ANOVA), matrix plots and correlation matrix.

4.7.1 Principal Component Analysis (PCA) of Dataset

The PCA was conducted in SCAN using the correlation matrix approach. As with the previous experiment the data evaluated was the change from the fresh lubricant, because this would allow for comparisons in the way the lubricant had degraded, rather that one lubricant had a different absolute value from another.

The PCA analysis of the Correlation Matrix is shown below.

Principal Component Analysis

Calculated from Correlation Matrix by SVD

Eigenvalue	2.076	1.372	0.853	0.697	0.002
Proportion	0.415	0.274	0.171	0.139	0.000
Cumulative	0.415	0.690	0.860	1.000	1.000

Eigenvectors

Variable	PC1	PC2	PC3	PC4	PC5
TGARV	-0.306	-0.315	-0.828	0.346	0.021
Visc40C	-0.547	-0.372	0.120	-0.502	-0.544
Visc100C	-0.621	0.326	0.008	-0.275	0.658
VI	-0.221	0.789	-0.156	0.182	-0.521
BN	0.415	0.183	-0.524	-0.720	0.006

Examination of the PCA output highlights that the first two Principal Components (PC) account for 69% of the variation in the data. This is considered to be slightly less than ideal, therefore interpretation of the results have to be treated with caution, due to 31% of the variation the data not being accounted for by the 1st two PC's.

The Principal Component Loading Plot for PC1 verses PC2 is shown in Figure 4.12.

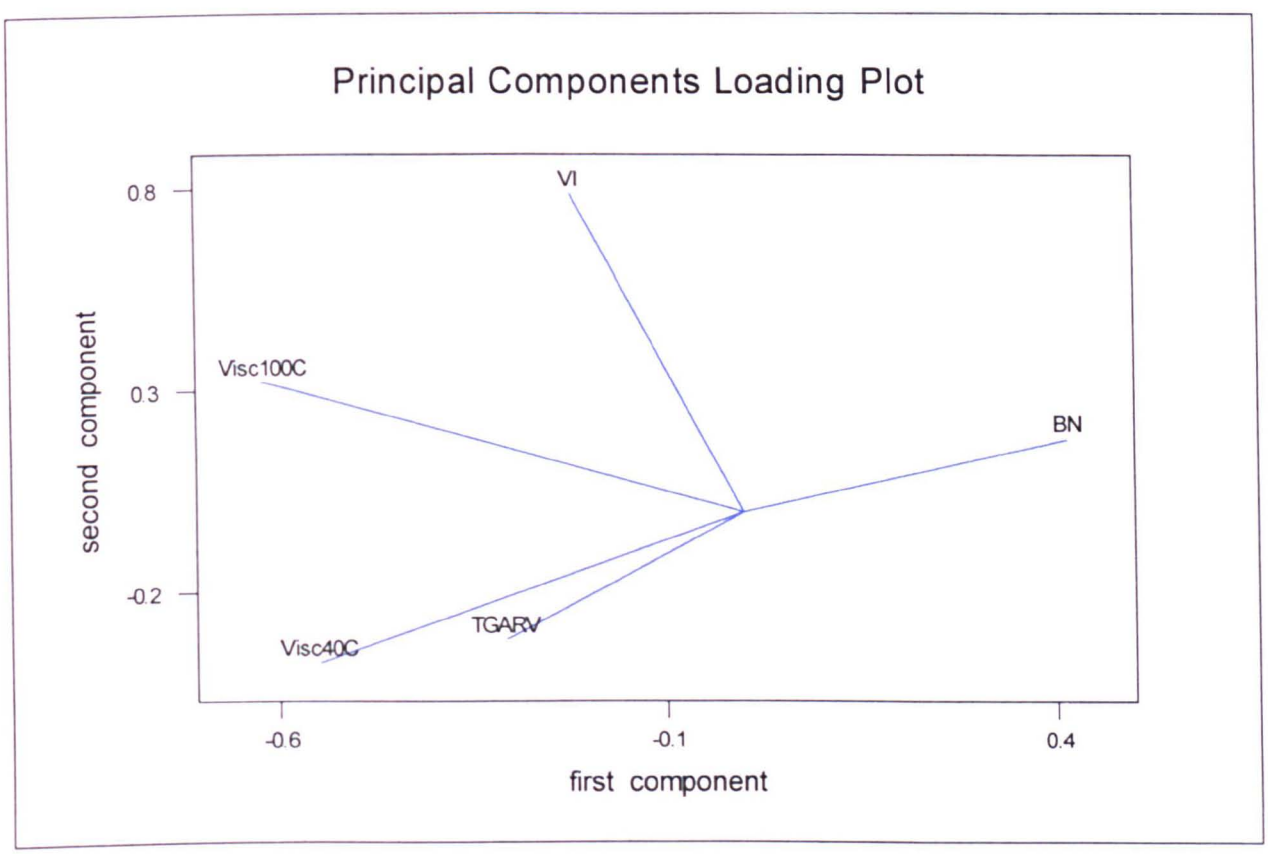


Fig. 4.12 Principal Component Loading Plot for PC1 verses PC2

Examination of the PC Loading Plots highlights that:-

PC1 is influenced by,

- BN in a positive direction.
- Visc100C, Visc40C and TGARV in a negative direction, although Visc100C and Visc40C have more of an effect.
- VI has little influence on PC1.

PC2 is influenced by,

- VI and Visc100C in a positive direction, although VI has more of an effect.
- Visc40C and TRGRV in a negative direction, although the effect is small.
- BN has little influence on PC2.

Interpretation of the PC loading plots is not an easy task, although it appears that PC1 is more influenced by the relationship between BN and viscosity changes. PC2 appears to be more influenced by the relationship between viscosity and relative volatility changes.

The Principal Component Score plot with respect to fuel sulphur content on the measured variables is shown in Figures 4.13.

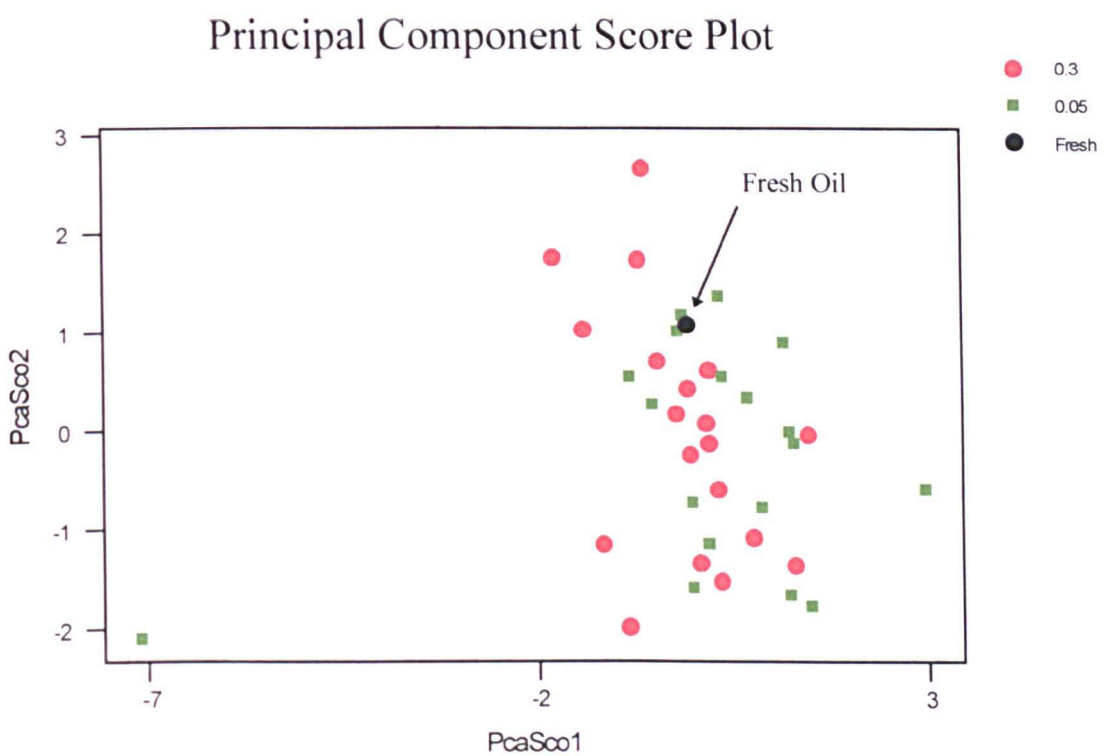


Fig. 4.13 Principal Component Score Plot for PC1 verses PC2 (with to respect fuel sulphur content)

Examination of Fig. 4.13 immediately highlights one point in the bottom left hand corner of the plot which stands out. This particular point has an extreme change in viscosity (a 89% increase at 40°C and a 42% increase at 100°C). This point has come from sampling position

3, from oil I (synthetic based) and was being run with a fuel sulphur content of 0.05%. This point is therefore being more noticeably effected by the sampling position and fuel sulphur content.

Apart from that particular point there are some possible patterns in the data in that more 0.3% sulphur fuel samples appear to the left of the fresh oil point. Also more 0.3% sulphur points appear above the fresh oil point.

The descriptive statistics, highlighting the effects of fuel sulphur content in terms of mean and standard deviation is shown in Table 4.4.

	MEAN VALUES				
Fuel Sulphur Content	TGARV	Visc40C	Visc100C	VI	BN
0.05%	0.105788	0.017711	-0.08328	-0.1234	-0.05674
0.30%	0.115304	-0.01186	-0.07189	-0.07973	-0.16933
	STANDARD DEVIATION				
Fuel Sulphur Content	TGARV	Visc40C	Visc100C	VI	BN
0.05%	0.164016	0.227039	0.147293	0.11616	0.135708
0.30%	0.111156	0.043303	0.081043	0.148894	0.216689

Table 4.4 Descriptive Statistics in Terms of Fuel Sulphur Content

Review of Table 4.4. highlights oils run with the 0.3% sulphur fuel have shown a much greater decrease in BN compared to oils run with a 0.05% sulphur content. Further review of Fig. 4.13 shows that points to the left hand side of the fresh oil point have shown a reduction in BN and very little change to volatility and viscosity, whilst points to the right hand side of the fresh oil point exhibit an increased BN, a reduced viscosity and an increased volatility. Points above the fresh oil point exhibit a reduced volatility and a increased viscosity, whilst points below the fresh oil point exhibit a increased volatility and a reduced viscosity.

This demonstrates that points to the left hand side of the fresh oil point have been effected by the formation of sulphur containing acids, as part of the combustion process, which is related to the sulphur level in the fuel. This explains why more points in this region have been run with the 0.3% sulphur fuel. Points above the fresh oil point have appeared to have

lost the 'light' ends of the base oil, which is being measured as a reduction in volatility and a increase in viscosity. This viscosity increase however may not purely be due to the loss of the 'light' ends of the base stock, it may also be due to the effectual increase in the concentration of the additives in the lubricant brought about by the loss of 'light' ends. The points below the fresh oil points have possibly been contaminated with fuel or subjected to shearing of longer chain molecules, because the volatility has increased and the viscosity has decreased.

The Principal Component Score plot with respect to sampling position on the measured variables is shown in Figures 4.14.

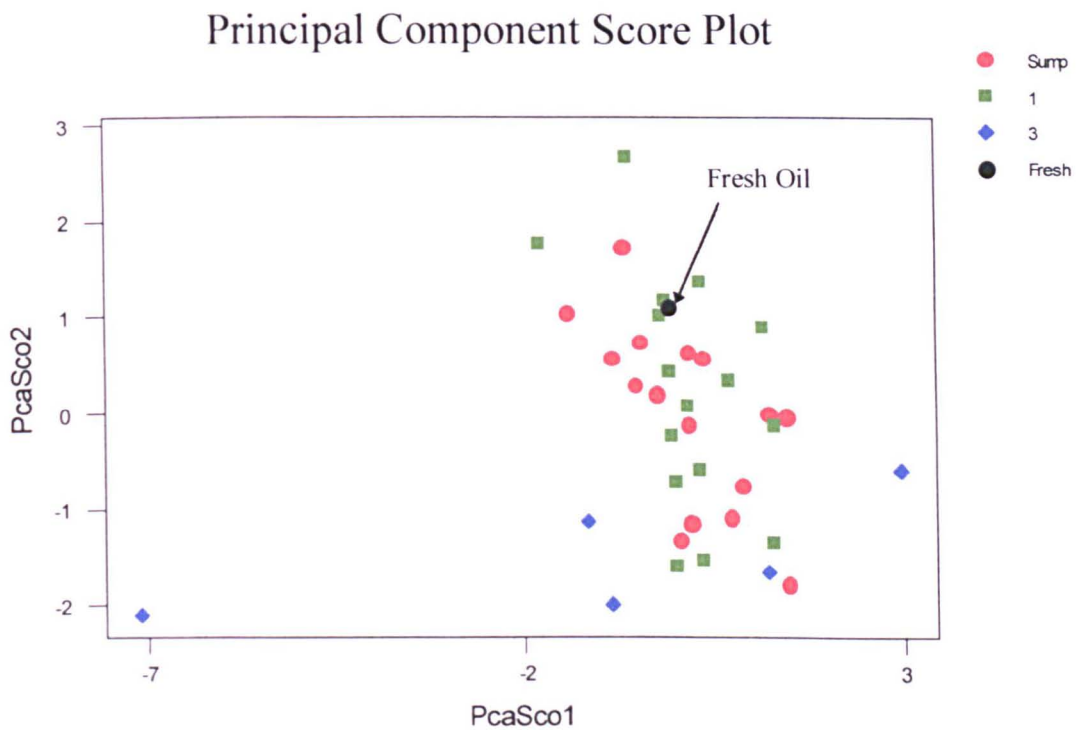


Fig. 4.14 Principal Component Score Plot for PC1 verses PC2 (with respect to sampling position)

Examination of Fig. 4.14 highlights that that the spread of points associated with the samples taken from the sump and Position 1 are very similar. This demonstrates that the lubricant degradation is similar for the sump oil and the oil sampled at Position 1. The samples taken from Position 3 however all appear below the Fresh oil point, highlighting that the samples have an increased volatility and a reduced viscosity, with respect to the

fresh oil point. If the point on the bottom left hand corner is excluded it can be seen that there are two points to the right of the fresh oil point, and two to the left. The points to the left have been run with 0.05% sulphur fuel, whilst the points to the left have been run with 0.3% sulphur fuel. As previously seen in Fig. 4.13, this highlights the effect of the increased sulphur fuel level on BN, because samples to the left have reduced BN's, whilst samples to the right gave increased BN's.

The Principal Component Score plot with respect to oil type on the measured variables is shown in Figures 4.15.

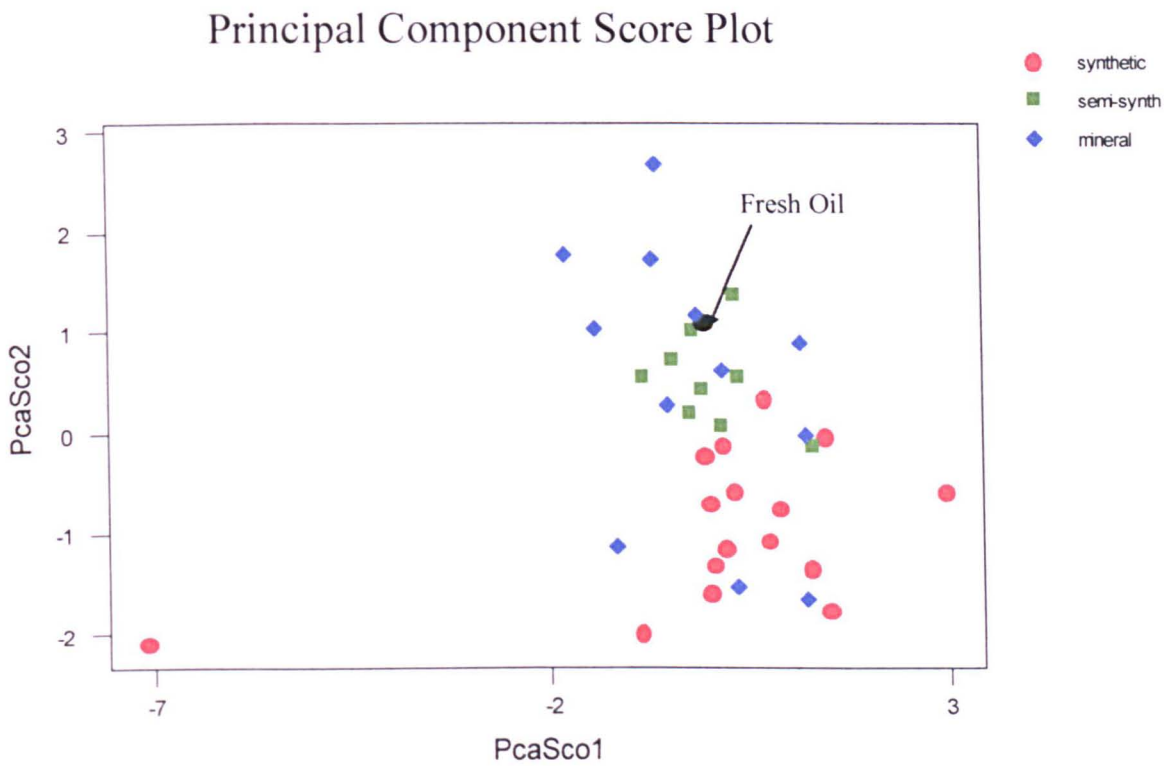


Fig. 4.15 Principal Component Score Plot for PC1 verses PC2 (with respect to oil type)

Examination of Fig 4.15 highlights some interesting trends in lubricant degradation with respect oil type. It can be seen that there appears to be partial separation of the groups in a vertical direction, with the synthetic lubricant being below the fresh oil point, the semi-synthetic lubricants are also below the fresh oil, but to a lesser extent, whilst the mineral

based lubricants are generally above the fresh oil point. Reviewing the previous findings the above demonstrates that the mineral based lubricants are more prone to volatility decreases and viscosity increases, whilst the semi-synthetic and synthetic based lubricants are more prone to volatility increases and viscosity decreases, however synthetic lubricants are more prone to this.

The Principal Component Score plot with respect to oil code on the measured variables is shown in Figures 4.16.

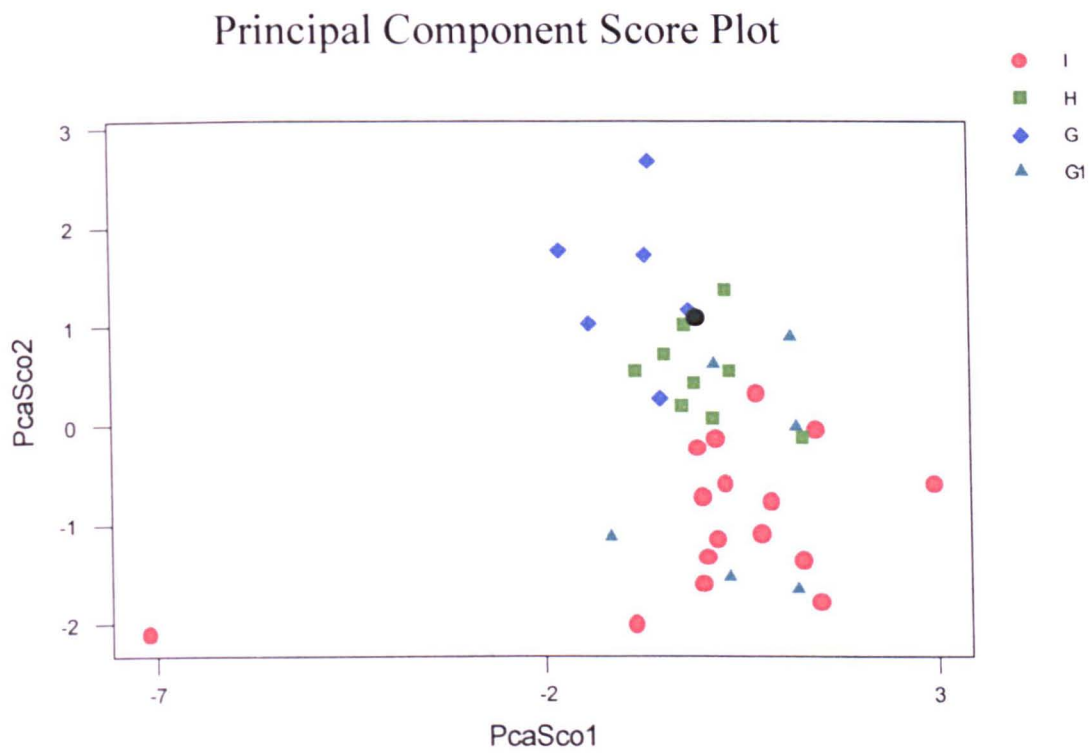


Fig. 4.16 Principal Component Score Plot for PC1 verses PC2 (with respect to oil code)

Examination of Fig. 4.16 also highlights an interesting trend, in that the mineral oil points (blue points) seen previously nesting within the synthetic oil points (red points) in Fig. 4.15 are in fact a different mineral based lubricant. It can therefore be concluded that oil G1 is performing more like a synthetic lubricant in terms of volatility and viscosity change.

4.7.2 ANOVA Analysis of Dataset

Analysis of Variance was carried out to examine the findings noted from the Principal Component Analysis. The ANOVA output is shown below.

General Linear Model

```
Factor      Type Levels Values
Oil Type    fixed      3 mineral semi-synthetic synthetic
Fuel        fixed      2 0.05 0.3
Position    fixed      3 1 3 Sump

Analysis of Variance for TGARV, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2    0.19333    0.18652    0.09326    5.95  0.006 SIG
Fuel         1    0.00107    0.00122    0.00122    0.08  0.782
Position     2    0.00040    0.00040    0.00020    0.01  0.987
Error       31    0.48576    0.48576    0.01567
Total       36    0.68056

Analysis of Variance for Visc40C, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2    0.04904    0.02908    0.01454    0.63  0.539
Fuel         1    0.00751    0.00267    0.00267    0.12  0.736
Position     2    0.14739    0.14739    0.07369    3.20  0.055
Error       31    0.71419    0.71419    0.02304
Total       36    0.91813

Analysis of Variance for Visc100C, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2    0.03462    0.03731    0.01866    1.29  0.290
Fuel         1    0.00090    0.00125    0.00125    0.09  0.770
Position     2    0.00449    0.00449    0.00224    0.16  0.857
Error       31    0.44824    0.44824    0.01446
Total       36    0.48825

Analysis of Variance for VI, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2    0.18724    0.15568    0.07784    6.32  0.005 SIG
Fuel         1    0.01446    0.00971    0.00971    0.79  0.381
Position     2    0.06283    0.06283    0.03141    2.55  0.094
Error       31    0.38153    0.38153    0.01231
Total       36    0.64606

Analysis of Variance for BN, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2    0.11208    0.13728    0.06864    2.89  0.070
Fuel         1    0.10597    0.15263    0.15263    6.43  0.016 SIG
Position     2    0.32185    0.32185    0.16093    6.78  0.004 SIG
Error       31    0.73553    0.73553    0.02373
Total       36    1.27543
```

The ANOVA analysis demonstrates that the significant effects (i.e. p-value less than 0.05) are:-

- TGA is significantly effected by Oil Type.
- Kinematic Viscosity at 40°C has no significant effects, although Sampling Position is only marginally non-significant.
- Kinematic Viscosity at 100°C has no significant effects.
- Viscosity Index is significantly effected by Oil Type, although Sampling Position is only marginally non-significant.
- BN is significantly effected by Fuel and Sampling Position, although Oil Type is only marginally non-significant.

These findings are demonstrated graphically in the main effects plots (Fig. 4.17 a to 4.17e).

Fig. 4.17a Main Effects Plot Highlighting the Percentage Change in Relative Volatility with Respect to Oil Type, Fuel Sulphur Level and Sampling Position

Fig 4.17a demonstrates that the significant effect is oil type.

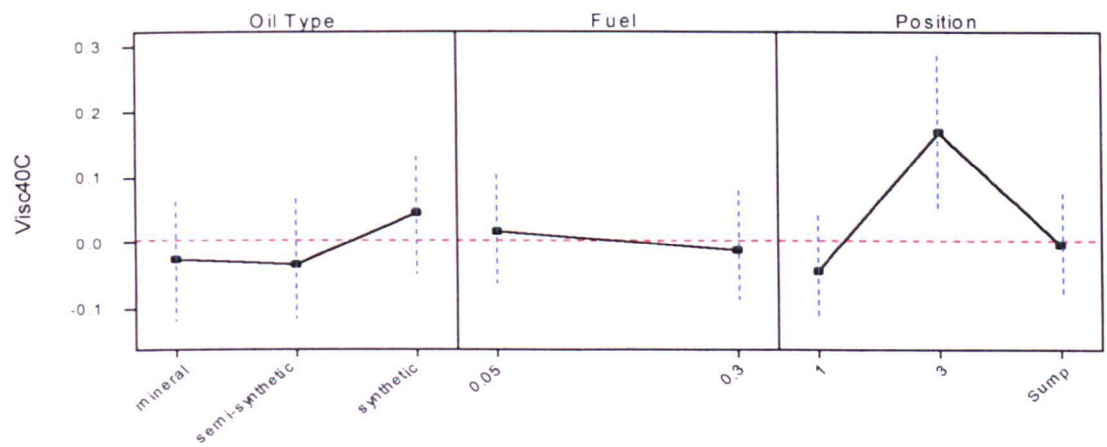


Fig. 4.17b Main Effects Plot Highlighting the Percentage Change in Viscosity at 40°C with Respect to Oil Type, Fuel Sulphur Level and Sampling Position

Fig 4.17b that there are no significant effects (although sampling position is only marginally non-significant).

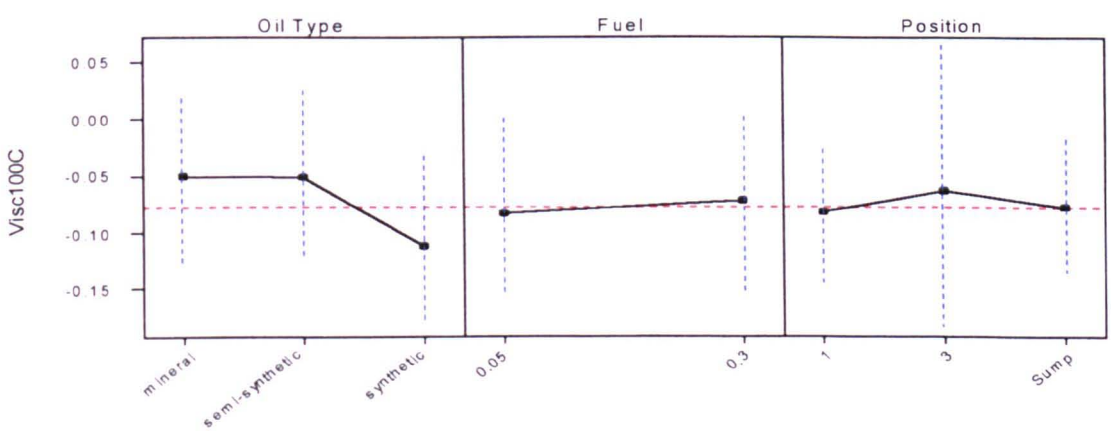


Fig. 4.17c Main Effects Plot Highlighting the Percentage Change in Viscosity at 100°C with Respect to Oil Type, Fuel Sulphur Level and Sampling Position

Fig. 4.17c that there are no significant effects.

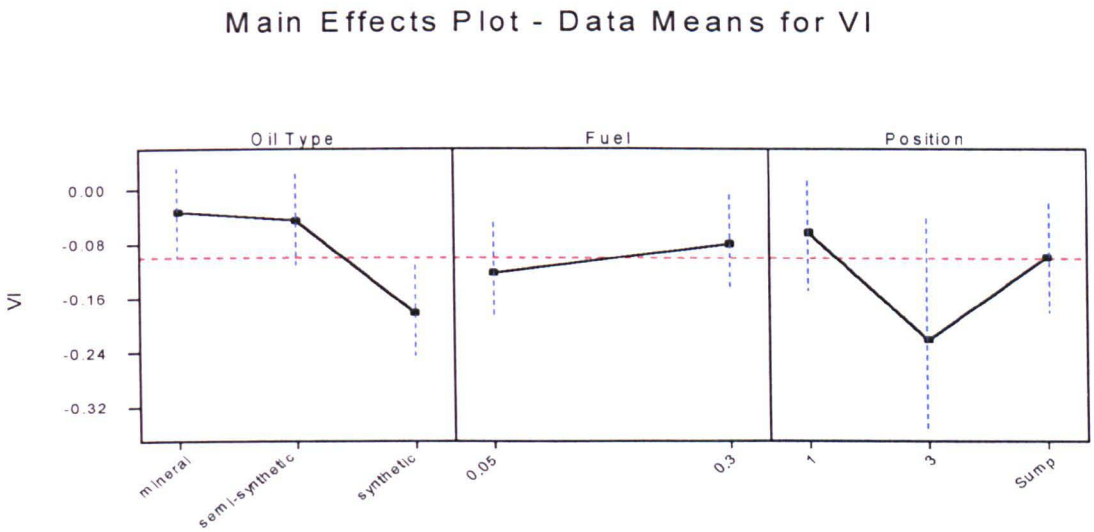


Fig. 4.17d Main Effects Plot Highlighting the Percentage Change in Viscosity Index with Respect to Oil Type, Fuel Sulphur Level and Sampling Position

Fig. 4.17d demonstrates that the significant effect is oil type.

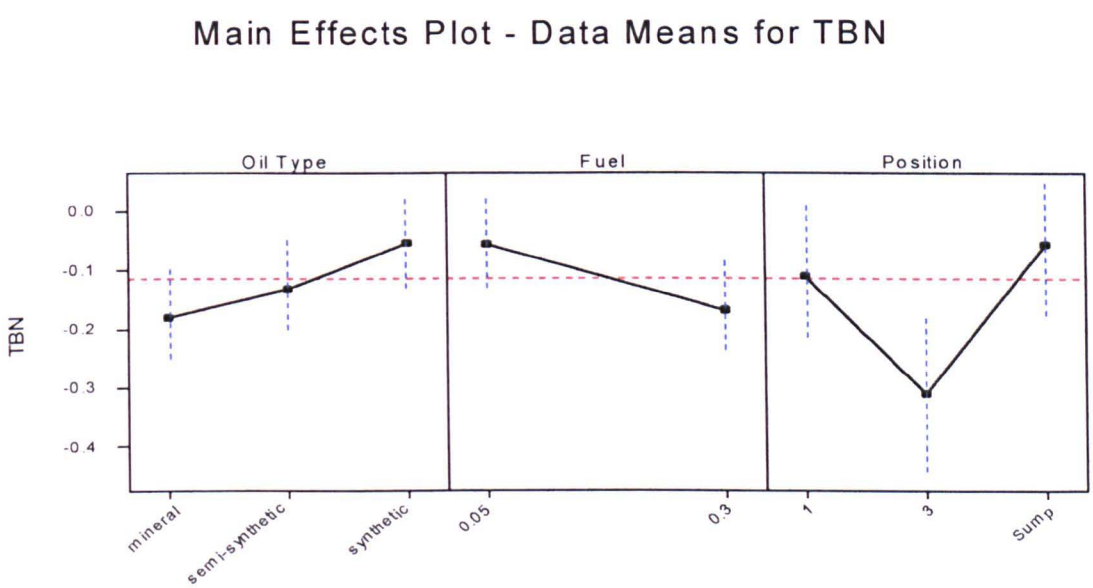


Fig. 4.17e Main Effects Plot Highlighting the Percentage Change in Base Number with Respect to Oil Type, Fuel Sulphur Level and Sampling Position

Fig. 4.17d demonstrates that the significant effects is fuel and sampling position.

4.7.3 Correlation's within the Dataset

The correlation's with the measured variables are examined by the matrix plot, Fig. 4.18.

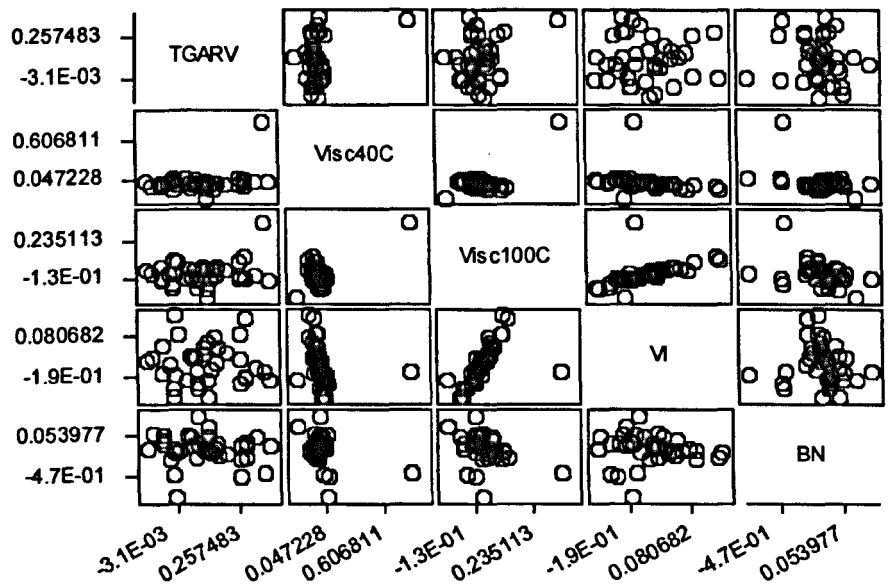


Fig 4.18 Matrix Plot of Measured Variables

Examination of the matrix plot demonstrates that there are potential linear trends according with the degradation of the lubricants. This is further demonstrated by the correlation matrix, shown below.

Correlations (Pearson)

	TGARV	Visc40C	Visc100C	VI
Visc40C	0.312			
	0.060			
Visc100C	0.246	0.650		
	0.142	0.000		
VI	0.011	-0.237	0.580	
	0.948	0.158	0.000	
BN	-0.105	-0.373	-0.373	-0.060
	0.536	0.023	0.023	0.722

Cell Contents: Correlation
P-Value

The correlation matrix highlights that there are significant linear relationships with Viscosity at 40°C and Viscosity at 100°C, with Viscosity at 40°C and Base Number, Viscosity at 100°C and Viscosity Index and Viscosity at 100°C and Base Number. There is also a marginally non-significant relationship with Relative Volatility at Viscosity at 40°C.

4.7.4 Conclusions from PCA and ANOVA

The conclusions from the PCA and ANOVA analysis are:-

1. The PCA analysis highlighted one point which showed a much greater change in viscosity. This oil sample came from position 3 and was run with 0.3% sulphur fuel.
2. Oils ran with a 0.3% sulphur content show more of a BN reduction than oils ran with 0.05% sulphur content. This is thought to be due to the increased chance of forming acidic sulphur containing by products of the combustion process.
3. The lubricant samples obtained from position 1 and sump samples show similar levels of degradation. Samples from position 3 show increased volatility and reduced viscosity when compared to fresh lubricants.
4. The different lubricant types show different degradation patterns. Synthetic oils show increased volatility and reduced viscosity, mineral based lubricants show reduced volatility and increased viscosity, whilst semi-synthetics behave like synthetics, but to a lesser extent.
5. Oil G1 (mineral based) behaves more like a synthetic lubricant than a mineral based lubricant in terms of volatility and viscosity change.
6. The ANOVA output highlights that significant changes occur with TGA (Oil type), Viscosity Index (Oil Type), and BN (Fuel Sulphur Content and Sampling Position).
7. The linear correlation's with the variables occur with Viscosity at 40°C and Viscosity at 100°C, with Viscosity at 40°C and Base Number, Viscosity at 100°C and Viscosity Index and Viscosity at 100°C and Base Number.

4.7.5 Examination of the Effects of Fuel Sulphur Content on Particle Size Distribution

The lubricant samples obtained from engine runs with differing fuel sulphur levels were analysed for particle size distribution. This experiment was to investigate the findings from other researchers[4.4, 4.5, 4.6] that the need to lower the fuel sulphur content in diesel fuels, due to emission regulations, was having a detrimental effect on performance on the dispersant/detergent additives in lubricants. It had been found that the particulate loading in lubricants was increasing as the sulphur content in the diesel fuels was decreased.

The experiment consisted of analysing lubricant samples from the sump and position 1 for Particle Size Distribution, by the method described in Chapter 2, Section 2.17. Samples from position 3 were not tested due to lack of sample size. Oil G (mineral based) and oil I (synthetic based) were tested for Particle Size Distribution.

Due to the findings from Chapter 2, Section 2.17, where it was found that as the sampling position moved up the piston there was an increase in particle size, the results obtained from the sump and position 1 have been represented separately. The results are presented in Fig 4.19a and 4.19b.

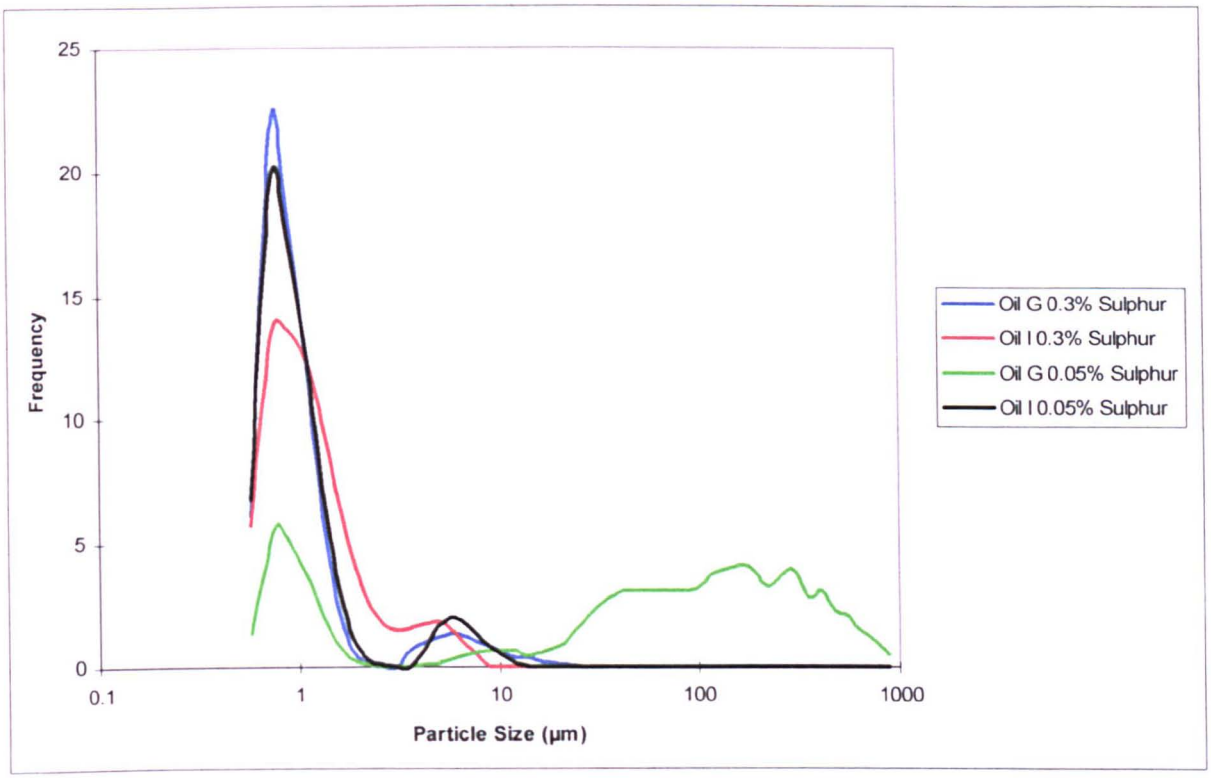


Fig. 4.19a Particle Size Distribution Obtained from Sump Samples using 0.3% and 0.05% Sulphur Diesel Fuel

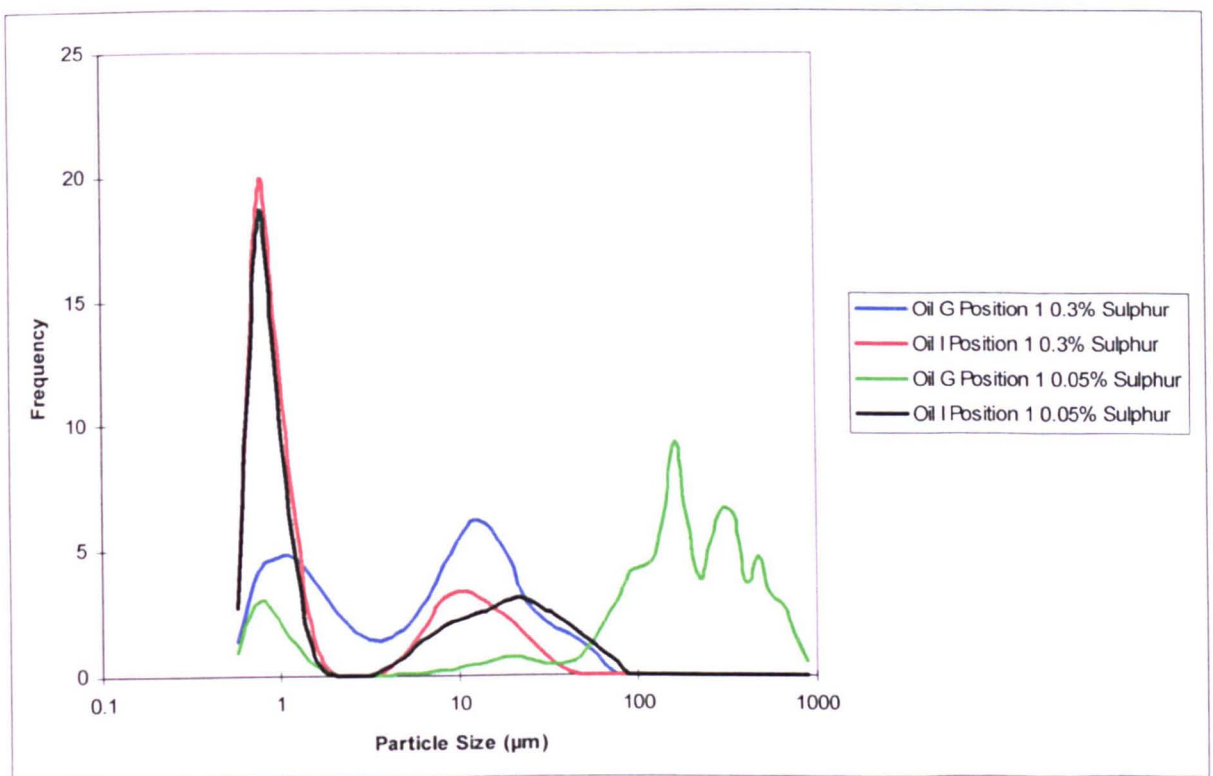


Fig. 4.19b Particle Size Distribution Obtained from Sampling Position 1 using 0.3% and 0.05% Sulphur Diesel Fuel

Examination of Fig. 4.19a highlights that the sump samples taken from engine runs using 0.3% sulphur have the majority of particles around 1μm in size, with a smaller distribution at 7μm. Sump samples taken from engine runs using 0.05% sulphur content diesel fuel show differences between the oil types. Oil I (synthetic based) shows little difference from the samples tested using 0.3%, whilst Oil G (mineral based) shows very little particles in the 1μm range with a large distribution centred around 100μm.

Examination of Fig. 4.19b highlights that both the fuel sulphur content and oil type have an effect on particle size distribution. Oil I had distributions at 1μm and 10μm when run with 0.3% sulphur fuel and distributions at 1μ and 20μm when run with 0.05% sulphur fuel. Oil G had distributions at 1μm and 15μm when run with 0.3% sulphur fuel and distributions at 1μ and 200μm when run with 0.05% sulphur fuel.

The particle size work has therefore demonstrated that running the CAT3406B with lower sulphur fuel increases the particle size distribution. It should be noted though that the increase in particle size distribution effected the mineral based lubricant to a far greater extend than the synthetic lubricant.

Reviewing the particle size distributions highlights that if lower sulphur diesel fuel is to be used in the CAT3406B then lubricants with a better dispersant/detergent additive pack need to be employed. The use of synthetic based lubricants appears to give better protection, in terms of reduced particle size with respect to fuel sulphur content, than mineral based lubricants.

4.8 Examination of the Effects of Piston Ring Pack and Fuel Sulphur Content on the Degradation of Various Compositions of Engine Lubricant

As with the previous experiment examining the effects of fuel sulphur content on lubricant degradation the engine speed was run at a constant speed of 1300rpm. Lubricant samples were also only taken from the sump and from behind the two compression rings (Position 1 and Position 3).

The suite of analytical tests applied to this experiment consisted of the measurement of:-

- Relative volatility by TGA. The TGA technique employed is described in Chapter 2, Section 2.15.
- Kinematic Viscosity at 40°C and 100°C. The Kinematic Viscosity technique employed is described in Chapter 2, Section 2.14.
- Calculation of Viscosity Index using the Viscosity at 40°C and 100°C as described in Chapter 2, Section 2.14.
- Base Number. The Kinematic Viscosity technique employed is described in Chapter 2, Section 2.16.

The data analysis techniques employed were Principal Component Analysis (PCA), Analysis of Variance (ANOVA), matrix plots and correlation matrix.

These particular experiments produced samples from position 1 and position 3 which contained very little lubricant (typically 0.3g for the 1991 rings and 0.1g for the 1994 rings for a 10 hour engine run). A full suite of analytical tests required approximately 1g of sample, therefore the oil samples from these positions could not be analysed by the all analytical tests. The reduction in sample size was thought to be due to the piston ring packs being used. The piston rings packs are discussed in more detail in Chapter 5. Only samples taken from the sump could be subjected to all analytical tests. Therefore the PCA and ANOVA analysis was only carried out on sump samples. It should be noted however that the engine runs times were limited to 10 hours, therefore the degradation of the sump samples would expected to be minimal. However it was thought interesting to see if any degradation trends could be noted after 10 hours, especially as typical engine tests last in excess of 100 hours.

4.8.1 Principal Component Analysis (PCA) of Dataset

The PCA was conducted in SCAN using the correlation matrix approach. As with the previous experiments the data evaluated was the change from the fresh lubricant, because this would allow for comparisons in the way the lubricant had changed, rather that one lubricant had a different absolute value from another one.

The PCA analysis of the Correlation Matrix is shown below.

Principal Component Analysis

Calculated from Correlation Matrix by SVD

Eigenvalue	2.233	1.820	0.580	0.365	0.002
Proportion	0.447	0.364	0.116	0.073	0.000
Cumulative	0.447	0.811	0.927	1.000	1.000

Eigenvectors

Variable	PC1	PC2	PC3	PC4	PC5
TGARV	0.375	0.416	0.753	-0.342	0.055
Visc40C	0.577	0.211	-0.497	-0.292	-0.538
Visc100C	-0.349	0.556	-0.380	-0.471	0.450
VI	-0.631	0.212	0.195	-0.118	-0.710
BN	0.077	0.654	-0.058	0.750	-0.014

Examination of the PCA output highlights that the first two Principal Components (PC) account for 81% of the variation in the data. It was thought that an acceptable amount of variation in the data was explained by the 1st two PC's, as only 19% of the variation the data not being accounted for by the 1st two PC's.

The Principal Component Loading Plot for PC1 verses PC2 is shown in Figure 4.20.

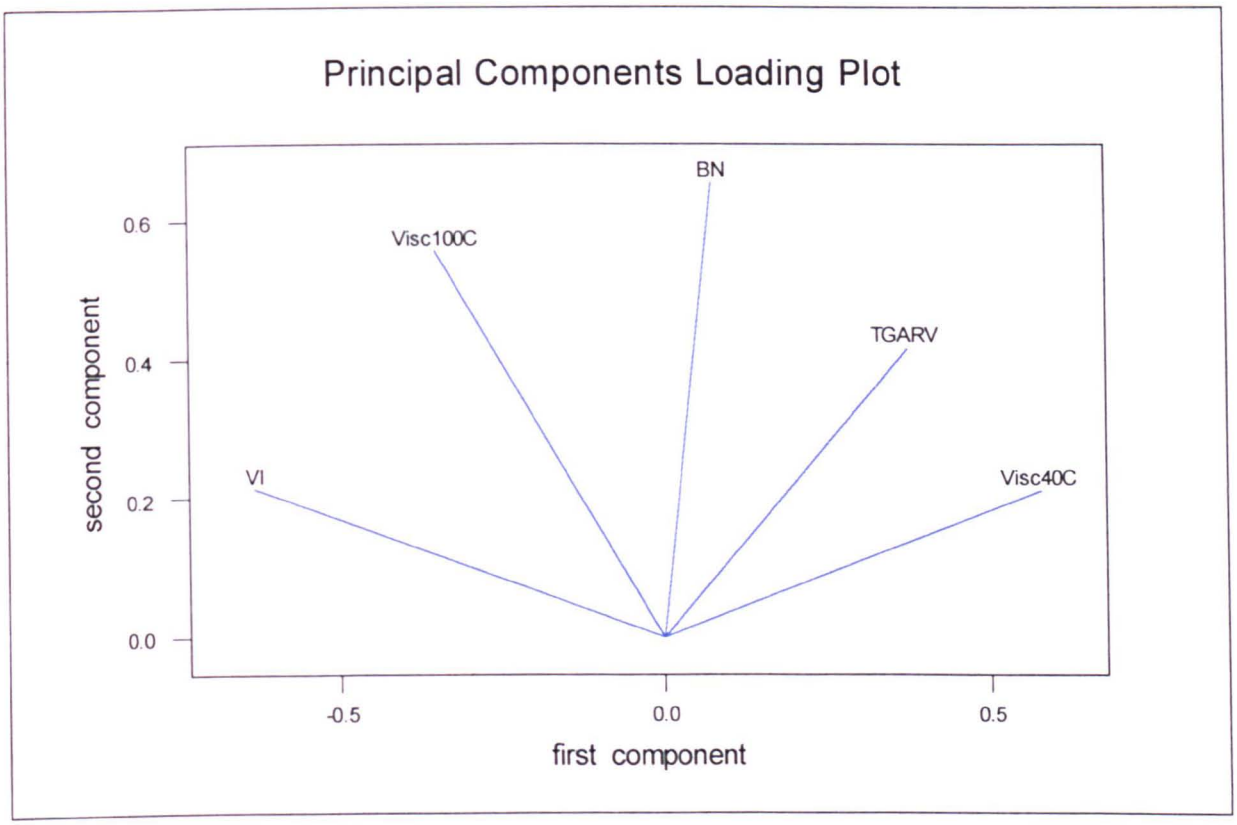


Fig. 4.20 Principal Component Loading Plot for PC1 verses PC2

Examination of the PC Loading Plots highlights that:-

PC1 is influenced by,

- Visc40C and TGARV in a positive direction, although Visc40C has more of an effect.
- VI and Visc100C in a negative direction, although VI has more of an effect
- BN has little influence on PC1.

PC2 is influenced by,

- BN, Visc100C and TGARV in a positive direction, although BN has more of an effect.
- VI and Visc40C has little influence on PC2.

It appears that PC1 is influenced by the relationship between viscosity and relative volatility (i.e. physical changes to the lubricant). PC2 is possibly more influenced by chemical changes in the lubricant, which is demonstrated by the inclusion of the changes in BN.

The Principal Component Score plot with respect to fuel sulphur content on the measured variables is shown in Fig. 4.21.

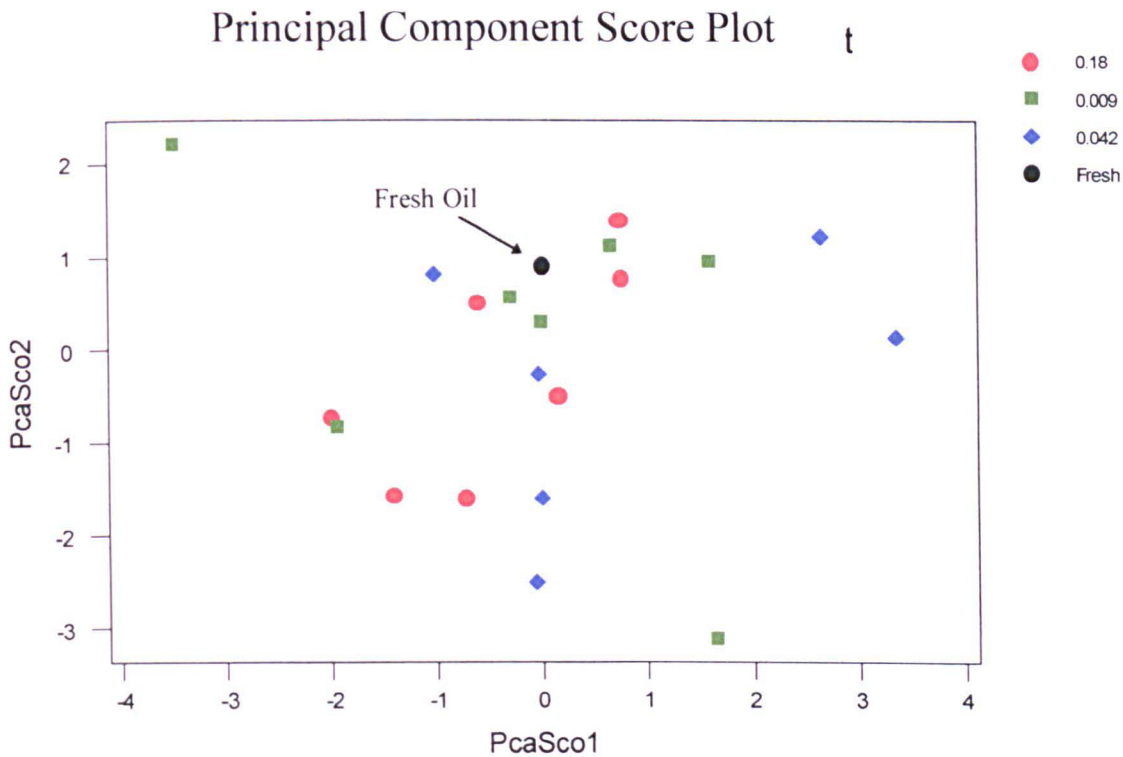


Fig. 4.21 Principal Component Score Plot for PC1 verses PC2 (with to respect fuel sulphur content)

Examination of Fig. 4.21 highlights that there does not appear to be any discernible pattern with respect to fuel sulphur content. This is contradictory to the findings shown in Fig. 4.13, however this may be due to the fact that only sump samples could be analysed.

The Principal Component Score plot with respect to piston ring pack type on the measured variables is shown in Figures 4.22.

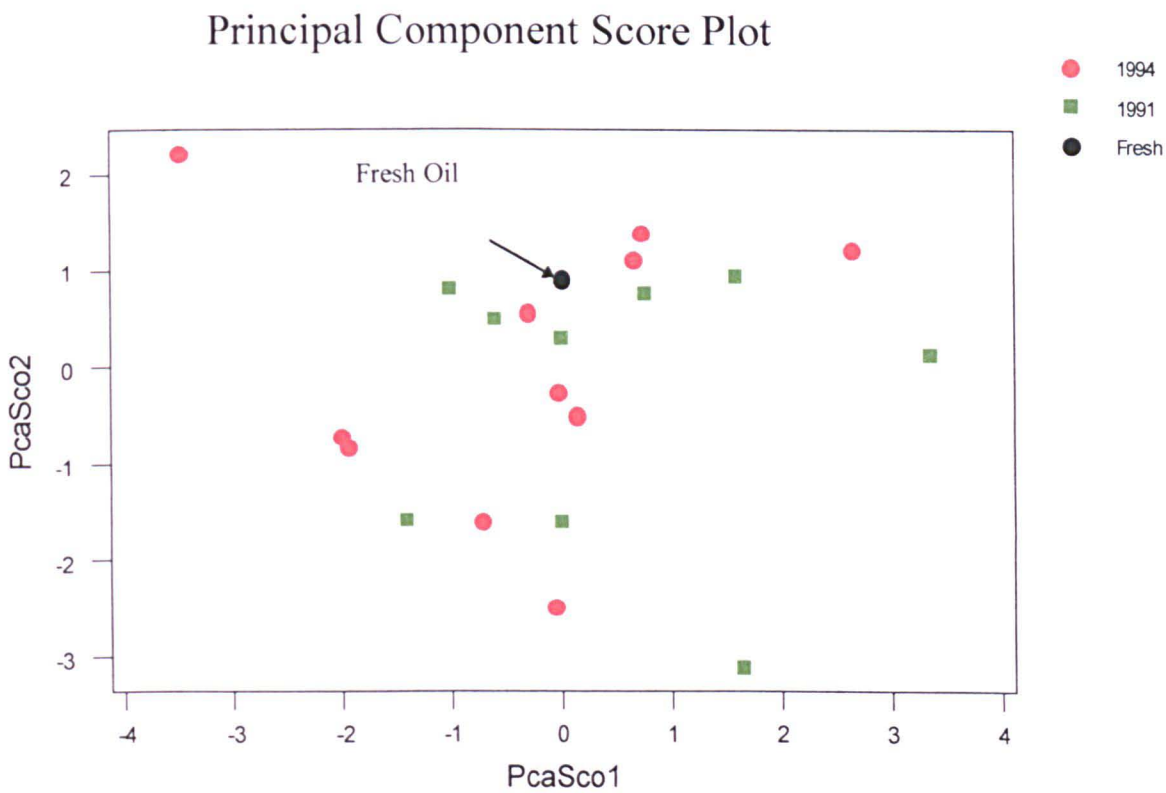


Fig. 4.22 Principal Component Score Plot for PC1 verses PC2 (with to respect piston ring pack type)

Examination of Fig. 4.22 also highlights that there does not appear to be any discernible pattern with respect to piston ring pack type. Further analysis of the use of different types of piston ring are reported in Chapter 5, however from this data it does appear that little difference in lubricant degradation can be noted from the different types. However once again this may be influenced by all samples being taken from the sump.

The Principal Component Score plot with respect to oil type on the measured variables is shown in Figures 4.23.

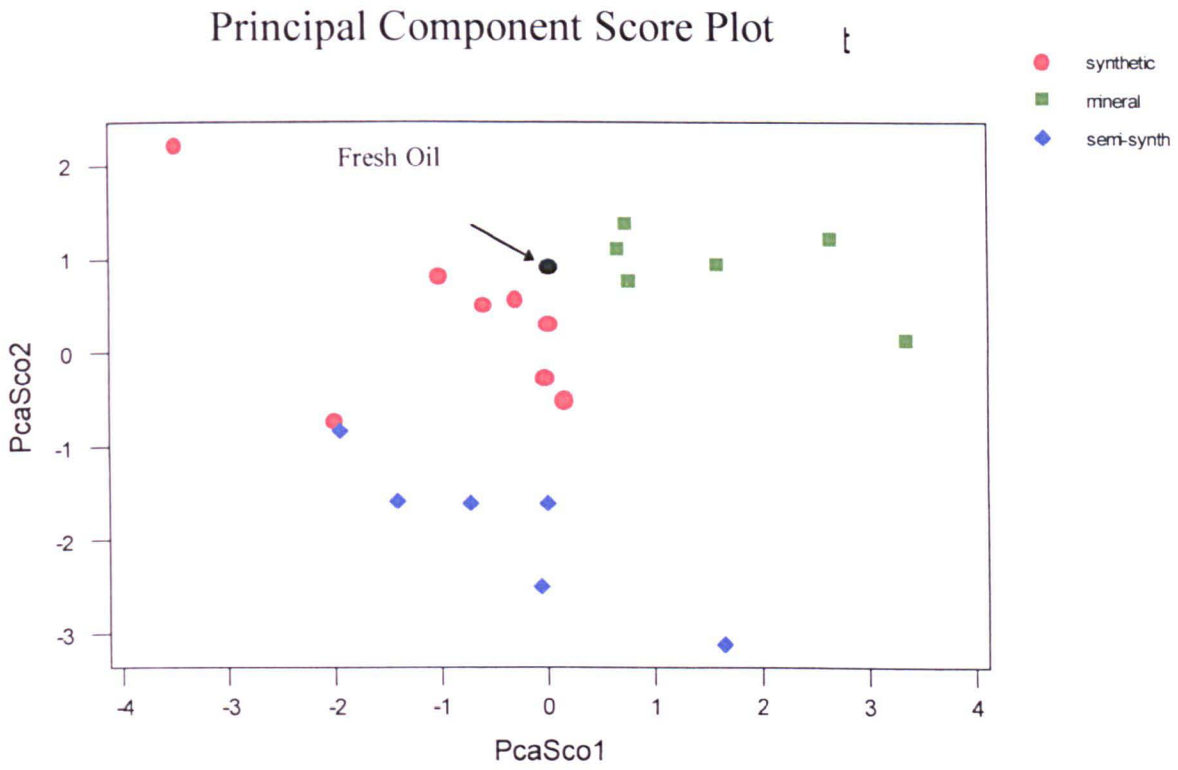


Fig. 4.23 Principal Component Score Plot for PC1 verses PC2 (with to oil type)

Examination of Fig 4.23 highlights an interesting trend with respect to lubricant type. It appears that the different oil types are forming clusters. The semi-synthetic lubricant gives points consistently below the Fresh oil sample, the mineral based lubricant gives points slightly above and to the right, whilst the synthetic based lubricant gives points slightly below and the to left of the Fresh sample.

The descriptive statistics for the dataset, with respect to lubricant type is shown in Table 4.5.

	MEAN				
Oil Type	TGARV	Visc40C	Visc100C	VI	BN
mineral	0.094388	0.022352	-0.02878	-0.06614	-0.02481
semi-synthetic	-0.02351	-0.06358	-0.05176	-0.0067	-0.3224
synthetic	0.018939	-0.08368	-0.02696	0.0625	-0.01505

	STANDARD DEVIATION				
Oil Type	TGARV	Visc40C	Visc100C	VI	BN
mineral	0.075561	0.018137	0.033744	0.06559	0.039934
semi-synthetic	0.033831	0.038087	0.046102	0.095196	0.142775
synthetic	0.033614	0.055543	0.036039	0.108183	0.060521

Table 4.5 Descriptive Statistics of the Dataset, with Respect to Oil Type

Examination of Fig. 4.22 in conjunction with Fig. 4.20 and Table 4.5 leads to the findings that:-

- the synthetic lubricant is giving the most change in Viscosity at 40°C, with very little change in the other measured variables,
- the semi-synthetic lubricant is giving the most change in Base Number, Viscosity at 100°C and a reduction in Relative Volatility, little change in the other measured variables,
- the mineral based lubricant is giving the most change in Relative Volatility and Viscosity Index, and little change in the other measured variables.

4.8.2 ANOVA Analysis of Dataset

Analysis of Variance was carried out to examine the findings noted from the Principal component analysis. The ANOVA output is shown below.

General Linear Model

```
Factor      Type Levels Values
Oil Type   fixed      3 mineral semi-synthetic synthetic
Fuel       fixed      3 0.009 0.042 0.18
Rings      fixed      2 1991  1994

Analysis of Variance for TGARV, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Oil Type     2   0.058684   0.058140   0.029070   12.21  0.001 SIG
Fuel         2   0.005054   0.004955   0.002478    1.04  0.379
Rings        1   0.000338   0.000338   0.000338    0.14  0.712
Error       14   0.033329   0.033329   0.002381
Total       19   0.097404
```

Analysis of Variance for Visc40C, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Oil Type	2	0.053729	0.050332	0.025166	20.85	0.000	SIG
Fuel	2	0.003892	0.003632	0.001816	1.50	0.256	
Rings	1	0.001392	0.001392	0.001392	1.15	0.301	
Error	14	0.016898	0.016898	0.001207			
Total	19	0.075911					

Analysis of Variance for Visc100C, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Oil Type	2	0.003487	0.002936	0.001468	0.95	0.412
Fuel	2	0.002091	0.001912	0.000956	0.62	0.554
Rings	1	0.001261	0.001261	0.001261	0.81	0.383
Error	14	0.021714	0.021714	0.001551		
Total	19	0.028553				

Analysis of Variance for VI, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Oil Type	2	0.075501	0.062218	0.031109	3.32	0.066
Fuel	2	0.021153	0.019356	0.009678	1.03	0.381
Rings	1	0.012083	0.012083	0.012083	1.29	0.275
Error	14	0.131028	0.131028	0.009359		
Total	19	0.239766				

Analysis of Variance for BN, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P	
Oil Type	2	0.52699	0.52308	0.26154	109.51	0.000	SIG
Fuel	2	0.00546	0.00547	0.00273	1.15	0.346	
Rings	1	0.00004	0.00004	0.00004	0.02	0.894	
Error	14	0.03343	0.03343	0.00239			
Total	19	0.56593					

The ANOVA analysis demonstrates that the significant effects (i.e. p-value less than 0.05) for the measured variables are:-

- TGA is significantly effected by Oil Type.
- Kinematic Viscosity at 40°C is significantly effected by Oil Type.
- Kinematic Viscosity at 100°C has no significant effects.
- Viscosity Index has no significant effects, although Oil Type is only marginally non-significant.
- BN is significantly effected by Oil Type.

These findings are demonstrated graphically in the main effects plots, Fig. 4.24 a to 4.24e.

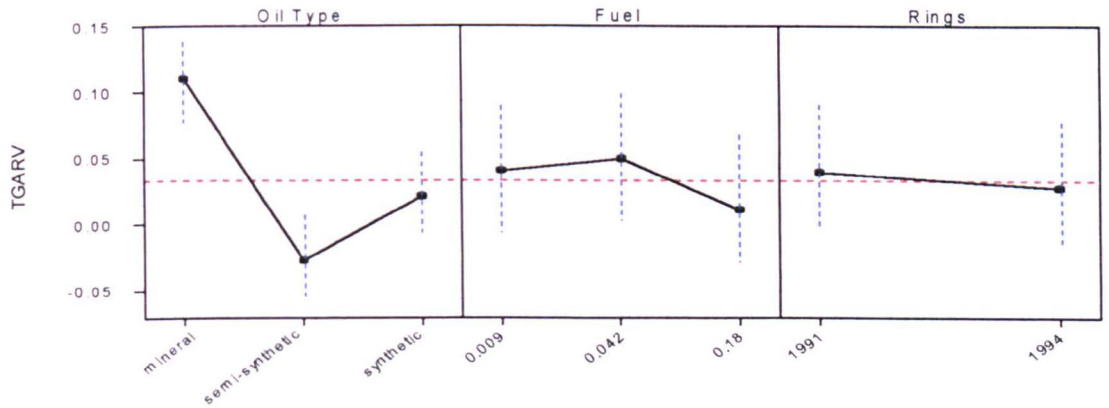


Fig. 4.24a Main Effects Plot Highlighting the Percentage Change in Relative Volatility with Respect to Oil Type, Fuel Sulphur Level and Piston Rings

Fig. 4.24a demonstrates that the significant effect is oil type.

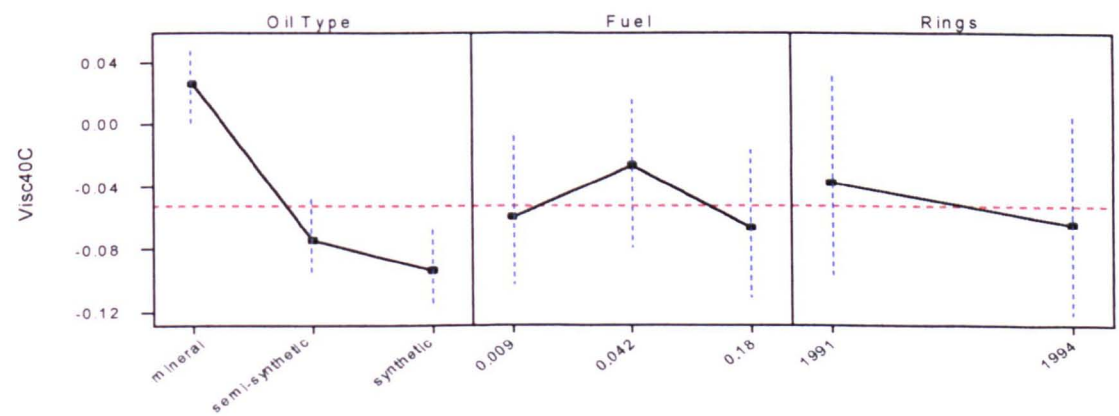


Fig. 4.24b Main Effects Plot Highlighting the Percentage Change in Kinematic Viscosity at 40°C with Respect to Oil Type, Fuel Sulphur Level and Piston Rings

Fig. 4.24b demonstrates that the significant effect is oil type.

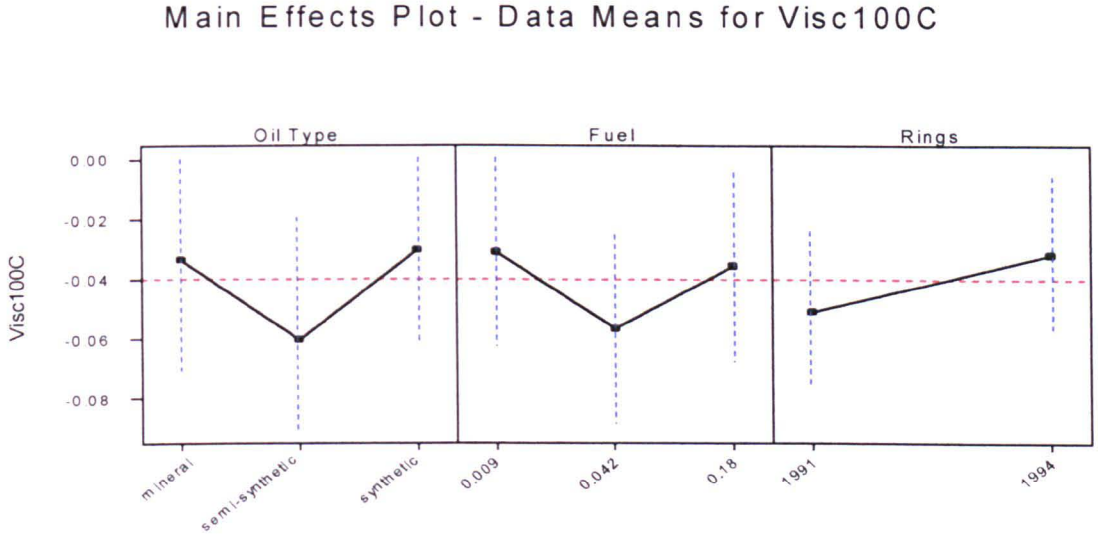


Fig. 4.24c Main Effects Plot Highlighting the Percentage Change in Kinematic Viscosity at 100°C with Respect to Oil Type, Fuel Sulphur Level and Piston Rings

Fig. 4.24c demonstrates that there are no significant effects.

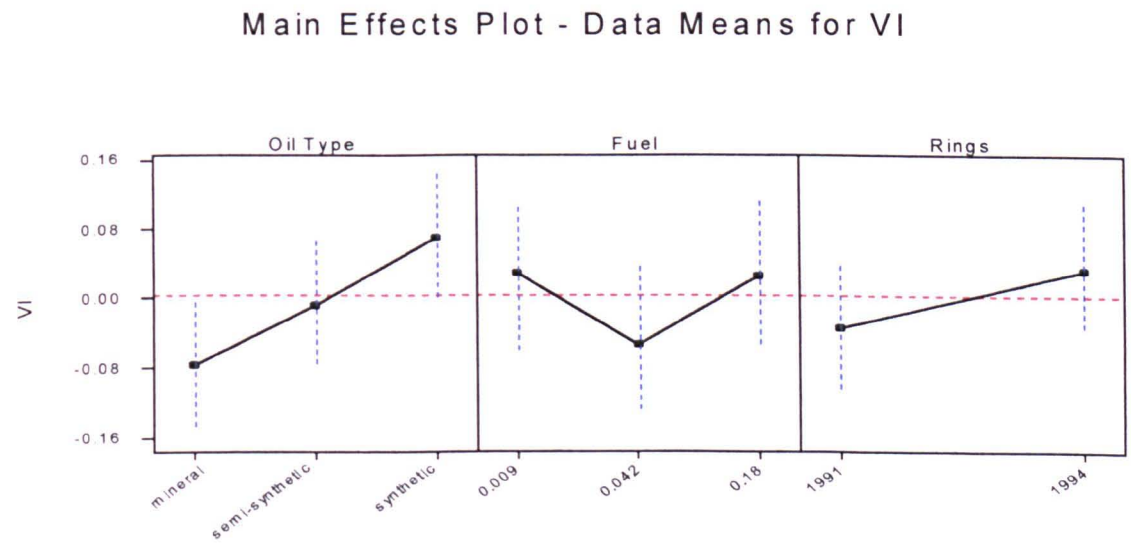


Fig. 4.24d Main Effects Plot Highlighting the Percentage Change in Viscosity Index with Respect to Oil Type, Fuel Sulphur Level and Piston Rings

Fig. 4.24d demonstrates that there are no significant effects (although oil type is only marginally non-significant).

Fig. 4.24e Main Effects Plot Highlighting the Percentage Change in Base Number with Respect to Oil Type, Fuel Sulphur Level and Piston Rings

Fig. 4.24e demonstrates that the significant effect is oil type.

4.8.3 Correlation's within the Dataset

The matrix plot of data is shown in Fig. 4.25.

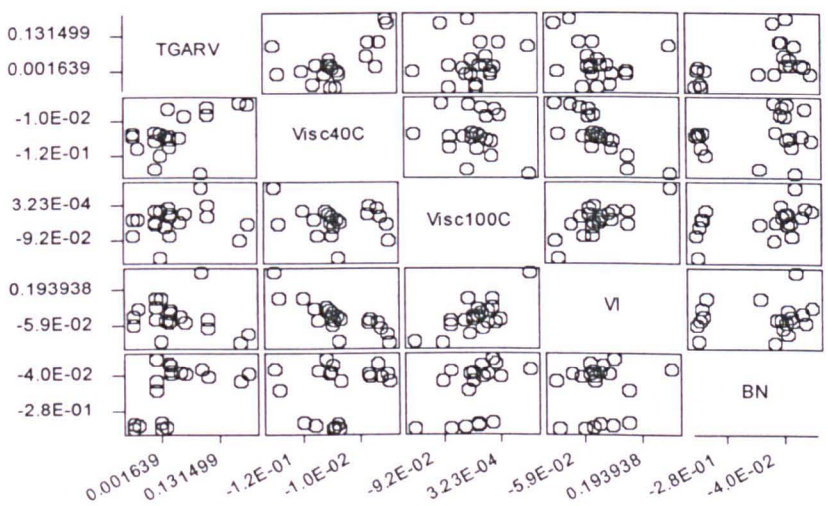


Fig 4.25 Matrix Plot of Measured Variables

Examination of the matrix plot demonstrates that there are potential linear trends according with the degradation of the lubricants. This is further demonstrated by the correlation matrix, shown below.

Correlations (Pearson)

	TGARV	Visc40C	Visc100C	VI
Visc40C	0.543			
	0.013			
Visc100C	0.090	-0.204		
	0.705	0.389		
VI	-0.274	-0.808	0.736	
	0.243	0.000	0.000	
BN	0.511	0.228	0.435	0.112
	0.021	0.335	0.055	0.639

Cell Contents: Correlation
P-Value

The correlation matrix highlights that there are significant linear relationships with Relative Volatility and Viscosity at 40°C, Relative Volatility and Base Number, Viscosity at 40°C and Viscosity Index, Viscosity at 100°C and Viscosity Index. There is also a marginally non-significant relationship with Viscosity at 100°C and Base Number.

4.8.4 Further Analysis of Effects of Fuel Sulphur Level and Piston Ring Type on Lubricant Degradation

Due to the lack of oil obtained from sampling positions 1 and 3 during the experiment a substantial part of the analytical analysis could not be carried out on the samples. Therefore the multivariate analysis reported in Chapter 4 Section 4.8 was carried out on samples obtained from the sump only. Due to the short engine run times (in the order of 10 hours) the lubricant obtained from the sump would not be expected to be significantly degraded, which possibly accounts for the findings noted in Fig.4.21 and 4.22. Further analytical analysis were carried out on the samples, however this covered the Relative Volatility Test only. Due to the univariate nature of the data the most appropriate analysis of the Relative Volatility data was by ANOVA.

The ANOVA analysis is shown below.

General Linear Model

```
Factor      Type Levels Values
Type       fixed      3 mineral semi-synthetic synthetic
Fuel       fixed      3 0.009 0.042 0.18
Rings      fixed      2 1991 1994
Position   fixed      3 P1  P3  Sump

Analysis of Variance for TGARV, using Adjusted SS for Tests

Source      DF     Seq SS     Adj SS     Adj MS       F      P
Type         2     3.0479     2.3547     1.1773    3.68  0.033 SIG
Fuel         2     0.0947     0.0386     0.0193    0.06  0.941
Rings        1     2.9254     2.9254     2.9254    9.16  0.004 SIG
Position     2    13.7610    13.7610     6.8805   21.53  0.000 SIG
Error       44    14.0584    14.0584     0.3195
Total       51    33.8874
```

Examination of the ANOVA output highlights that Sampling Position has the most significant effect on the change in Relative Volatility. This is followed by piston ring pack type and oil type.

The ANOVA output is shown graphically in the Main Effects plot, Fig. 4.26.

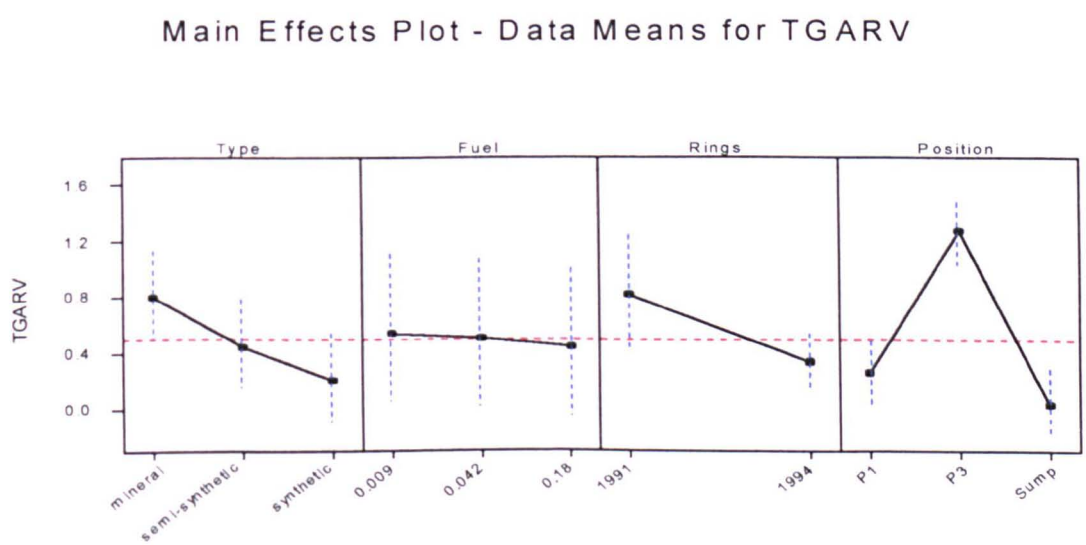


Fig. 4.26 Main Effects Plot Highlighting the Percentage Change in Relative Volatility with Respect to Oil Type, Fuel Sulphur Level, Piston Ring Pack Type and Sampling Position

Fig. 4.26 demonstrates that the largest effect on volatility is the sampling position on the piston, with position 3 providing a large positive increase in volatility. Fig. 4.26 also highlights that the 1994 piston rings produce less of a volatility increase than the 1991 rings. The 1991 and 1994 rings are discussed in more detail in Chapter 5, however one possible explanation for the lower volatility increase with the 1994 rings is that more lubricant was consistently obtained from positions 1 and 3 using the 1994 rings. The 1991 rings would, therefore, subject a smaller amount of lubricant to the extreme conditions associated with the piston ring zone and for a longer length of time. This would increase the chances of shearing the lubricant, and thus increasing the volatility.

Fig. 4.26 also demonstrates that fuel sulphur content has no effect on volatility.

Finally Fig.4.26 demonstrates, as expected, that the change in volatility follows the order of mineral lubricants > semi-synthetics lubricants > synthetics lubricants.

4.8.5 Conclusions for PCA and ANOVA Analysis

The conclusions from the PCA and ANOVA analysis are:-

1. The PCA analysis demonstrated that fuel sulphur content and piston ring pack type had no effect on lubricant degradation. However the PCA analysis could only be carried out on sump samples, which could be the reason for these findings.
2. The PCA analysis also demonstrated the lubricant type did have an effect on lubricant degradation. The synthetic lubricant gave most change in Kinematic Viscosity at 40°C, the semi-synthetic based lubricant gave most change in BN and Kinematic Viscosity at 100°C, whilst the mineral based lubricant gave the most change in relative volatility.
3. ANOVA analysis of the dataset used for the PCA analysis demonstrated that the only significant factor was oil type.
4. The correlation matrix of the PCA dataset demonstrated that linear correlation's existed for Relative Volatility and Viscosity at 40°C, Relative Volatility and Base Number, Viscosity at 40°C and Viscosity Index, Viscosity at 100°C and Viscosity Index.

5. Further ANOVA analysis based purely on relative volatility results demonstrated that
    - the most significant effect on volatility was sampling position, with samples from position 3 giving the most change,
    - fuel sulphur content had no effect on volatility,
    - the 1994 piston ring pack show less of a volatility change than the 1991 piston ring pack.

CHAPTER 5

Comparison of Piston Ring Pack Design on Lubricant Mass and Lubricant Transport

CHAPTER 5 Comparison of Piston Ring Pack Design on Lubricant Mass and Lubricant Transport

This Chapter examines the effects of piston ring pack design on lubricant mass, with particular emphasis placed on running under engine operating conditions of low speed and off load. The Chapter also discusses differences noted in lubricant transport with repect to piston ring pack design.

5.1 Comparison of Piston Ring Pack Performance - Introduction

The effect of running a Caterpillar 3406B for long periods under a regime of low speed and no load were investigated during this study. This work simulated the conditions an that engine would be subjected to if it were to be used as a standby generator. In these circumstances an engine would face long periods of running at low speeds under no load, and then short periods at high speeds under load. Consultation with engine manufactures found that problems were encountered with engines running under these conditions. It had been found that liquid lubricant was seen dripping from the exhaust. When the engine speed was increased and a load applied, clouds of blue/black smoke were then emitted from the exhaust. This phenomenon is called slobbering.

The phenomenon described above was thought to be due to the running conditions the engine was being subjected to. A low speed/no load running condition would mean the engine running at lower operating temperatures than would be encountered with 'normal' operating conditions. At 'low' operating temperatures it is probable that piston expansion into the cylinder bore would be less than intended. Therefore, more lubricant would be allowed to enter the combustion chamber with the possibility that some of this lubricant could be emitted via the exhaust port in liquid form. If the engine was suddenly placed under conditions of higher speed and load it is expected that this lubricant would be burnt, and because lubricant is a poor 'fuel' it does not burn cleanly. Therefore it would be expected that the characteristic blue/black smoke of burning lubricant would be seen exiting from the exhaust.

Experiments were carried out to monitor the amount of lubricant in the piston ring pack of the Caterpillar 3406B using the conditions of low speed and no engine load. Two different piston ring pack designs were used, so a comparison of piston ring pack performance versus lubricant mass in the piston ring zone could also be gained. The two ring pack configurations investigated were US Emission control ring packs dating from 1991 and 1994.

5.1.1 Comparison of Ring Pack Design for US Emission Control Ring Packs dated 1991 and 1994

The two ring packs were essentially of the same design, with a 'keystone' top ring, a 'stepped' second ring and a double 'knife-edged' oil control ring (ref: Chapter 1, Section 1.4). Little differences were noted with both the oil control ring and the second ring of both sets of ring packs, however a significant difference with the top rings were noted. The measured width of the 1994 rings were less than the 1991 rings, however the overall ring diameter remained the same. The measured ring widths are shown in Table 5.1. It was also noted that the 1994 rings were harder to compress than the 1991 rings, which was especially apparent when trying to compress the piston into the cylinder bore. The 1994 rings were therefore pushing harder against the cylinder bore wall. The effect of the rings pushing against the cylinder bore wall, was termed 'bore wall loading', with the 1994 rings having a higher bore wall loading than the 1991 rings. The actual difference between the 'bore wall loading' was not quantified during the experimentation.

Ring Pack	Width of 2nd Compression Ring	Width of 1st Compression Ring
Pre-1991 US Emission Control	5.5mm	5.5mm
1991 US Emission Control	5.5mm	5.4mm
1994 US Emission Control	5.5mm	5.3mm

Table 5.1 Measured Widths of 1st and 2nd Compression Rings Used in Caterpillar 3406B

A higher bore wall loading of the top ring, at its simplest, would have the effect of making the piston ring sit tighter between the piston and the cylinder bore wall interface. It is believed that this would remove more lubricant from the cylinder bore wall.

Previous research had demonstrated that piston ring design could effect wear[5.1], oil comsumption[5.2, 5.3, 5.4, 5.5, 5.6], and therefore the noted differences in piston ring design for the 1991 and 1994 rings were thought to be significant.

5.2 Experiments Undertaken with the Piston Ring Packs

Experiments were carried out by running the engine at the desired engine speed, with no load applied to the engine (i.e. the water brake not applied during engine runs). A minimum amount of water was allowed to flow through the engine to eliminate engine overheating.

The sampling piston used during these experiments allowed samples to be taken from both the second ring (position 1) and the top ring (position 3) simultaneously. Direct comparison of oil mass flow could therefore be obtained from both of the compression rings. Lubricant samples were taken at frequent intervals from both sampling positions, so that oil mass flow could be monitored over time. Typical engine run times were 10 hours. The oil mass measurement technique was the same as described in Chapter 2 (Section 2.6), with oil samples taken every 15 to 30 minutes.

5.3 Results Obtained from the Oil Mass Flow Experiments from the 1991 and 1994 Ring Packs

Initial findings from the 1991 rings showed complex results, Fig 5.1. The graph represents the mean oil mass flow at position 1 and 3 over three 10 hour engine runs.

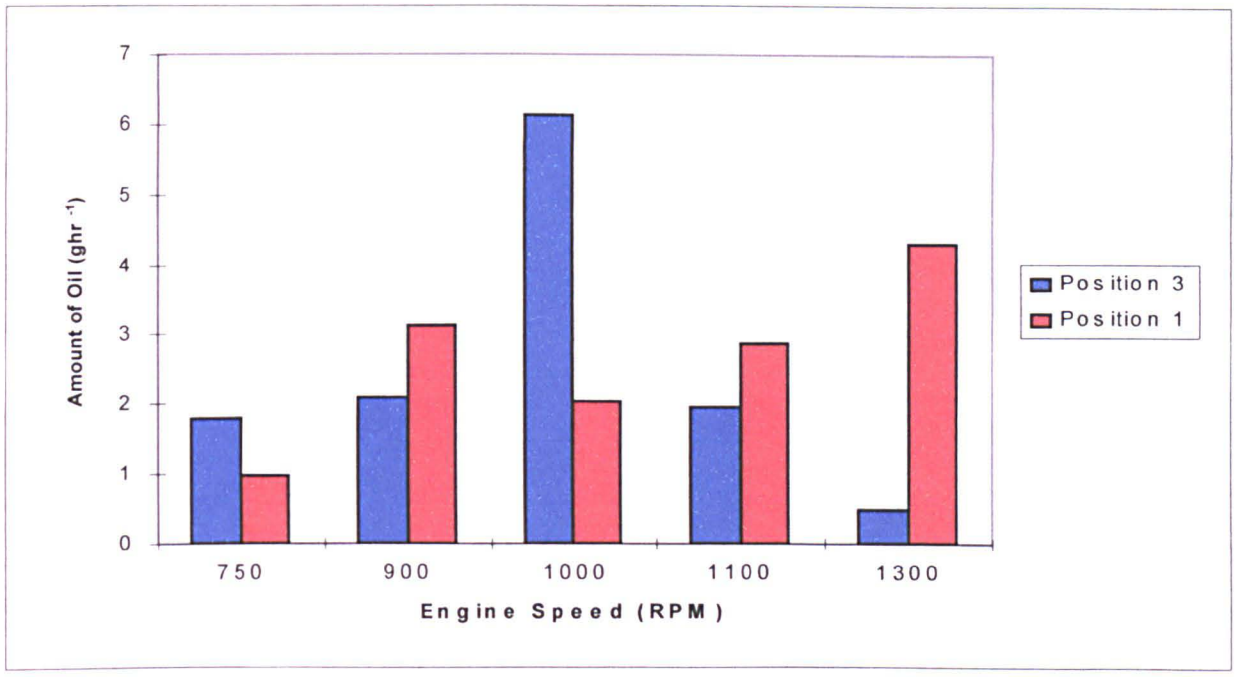


Fig. 5.1 Comparison of Oil Mass Flow Obtained for the 1991 US Emission Control Piston Rings under Engine Running Conditions of Low Speed/Off Load

At both 750 and 1000 RPM more lubricant was obtained from position 3 (top ring) than position 1 (2^{nd} ring). This showed an unexpected trend, where it appeared that oil flow was not being controlled in conventional terms. Expected oil control would show more oil being sampled from position 1 compared to position 3, because position 1 is further down the piston. It should be noted that piston ring lubricant flows are usually considered at a 'high' load level and at engine speeds above 1300 RPM. It can be argued that piston ring pack lubricant models are not applicable at low speed and no load conditions due to the reduced gas flows that the ring pack would be subjected to. The results obtained may, therefore, be considered to be 'normal' under these running conditions.

A closer examination of the oil mass flow obtained from one particular engine run (at 1000 RPM), Fig 5.2, showed that the oil flow from position 3 was consistently higher than position 1. From this particular experiment it can be seen that the oil mass from position 1 was almost consistent at 0.1g every 30 minutes, with the oil mass obtained from position 3 varying between 0.5 to 1.3g. It should be noted that the results shown in Fig. 5.2 were typical of results from these experiments, however a degree of variation was noted during

the experimentation. It was consistently demonstrated that there was a propensity for more lubricant to be sampled from position 3 than position 1, without any apparent cause.

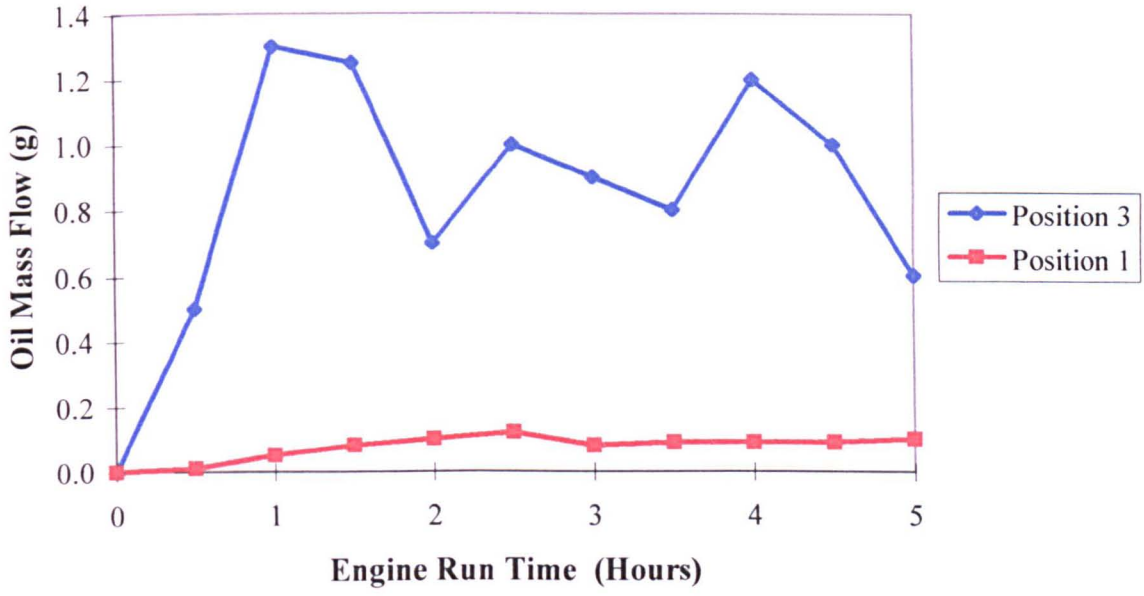


Fig. 5.2 Comparison of Oil Mass Flow Obtained from Sampling Positions 1 and 3 of the 1991 US Emission Control Piston Ring Under Low Speed/No Load Running Conditions (Engine Speed 1000 RPM)

5.3.1 Blocked Air Filter

During a routine engine strip down it was noticed that the air intake filter was becoming blocked. The air filter was replaced, and the oil mass flow experiments continued. The results obtained after replacement of the air intake filter were noticeably different than that obtained previously. Fig 5.3 shows the oil mass flow obtained from the 1991 piston rings pre and post air intake filter change.

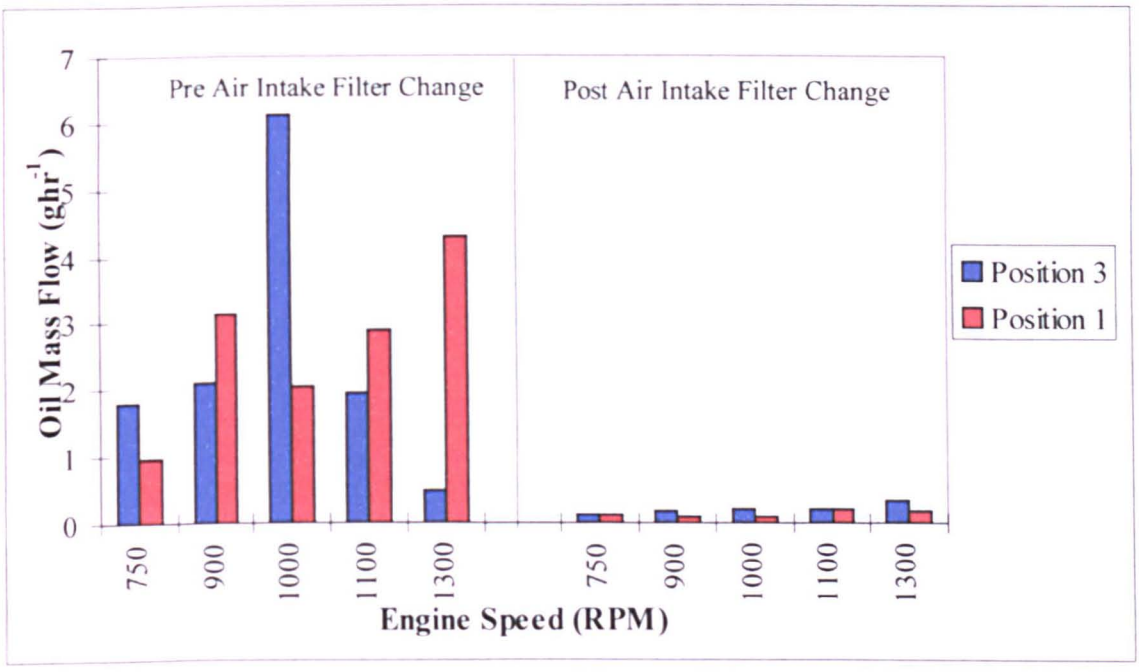


Fig. 5.3 Comparison of Oil Mass Flow from 1991 Rings Pre and Post Air intake Filter Change

The results showed significantly less oil being obtained from the sampling system post air filter change. After extensive investigations into this unexpected behaviour it was found that an almost completely blocked air filter could induce more lubricant into the ring pack in two ways.

1. By allowing cooling oil from the turbo charger into the combustion chamber. This was due to the 'o-ring' seal of the turbo charger requiring positive pressure to produce an oil-tight seal, which would only be achieved if the air filter remained unblocked. It was discovered that a blocked air filter produced a reduced pressure when compared to an unblocked filter, which could potentially provide an ineffective oil seal in the turbocharger. This would effectively draw lubricant from the turbocharger into the combustion chamber. This lubricant could potentially be transported to sampling positions on the piston.

2. By losing pressure at either piston ring position. It was thought that a blocked air intake filter could affect the gas flow pressure behind the piston rings, which would have the effect of producing less force behind the piston rings. This would result in the rings not

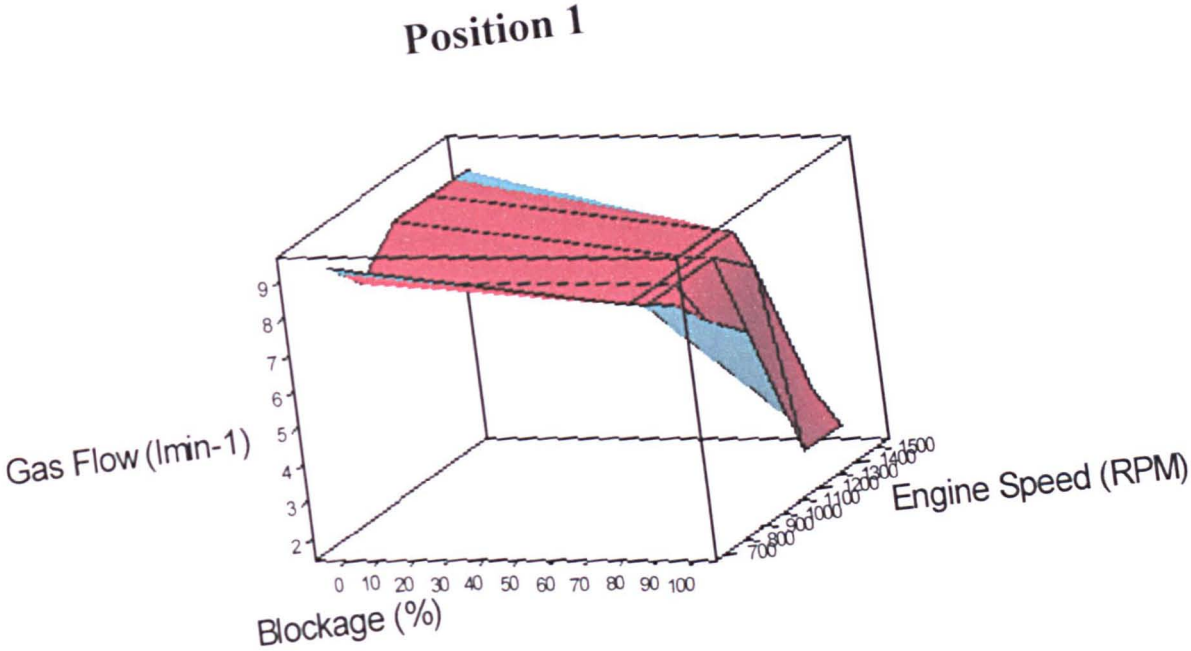
producing an tight oil seal on the piston/cylinder bore wall interface. If this occurred an increased amount of lubricant could pass from the sump into the piston ring pack, and thus be sampled.

It was decided to investigate the potential for loss of piston ring pressure, as an air filter became blocked, by measuring the gas flow at the two piston ring positions. An experiment was set up using a new air filter which was then artificially 'blocked' by covering the air intake holes by an increasing amount. The gas flows were measured at 0% to 97% blockage, at engine speeds of 750, 900, 1100, 1300 and 1500 RPM, and with the engine running both off and on load. The gas flow measuring method as described in Chapter 2 Section 2.6 was used.

The results showed that as the filter became blocked there was a loss of gas flow at position 3 (approximately 30%), however the decrease was much larger at position 1 (approximately 80%). Fig 5.4a and 5.4b highlights the data obtained with the engine running off and on load.

The data obtained demonstrates that running of the engine with the air filter blocked up to 90% has little effect on the gas flows at either sampling positions, however at a 97% blockage noticeable changes in gas flow were encountered. As the engine speed increased it became apparent that the gas flow was decreasing behind the piston rings.

Although subtle differences were noted between the data obtained on, and off, load the general trend of a large decrease in gas flow at position 1 was true for both sets of running conditions.



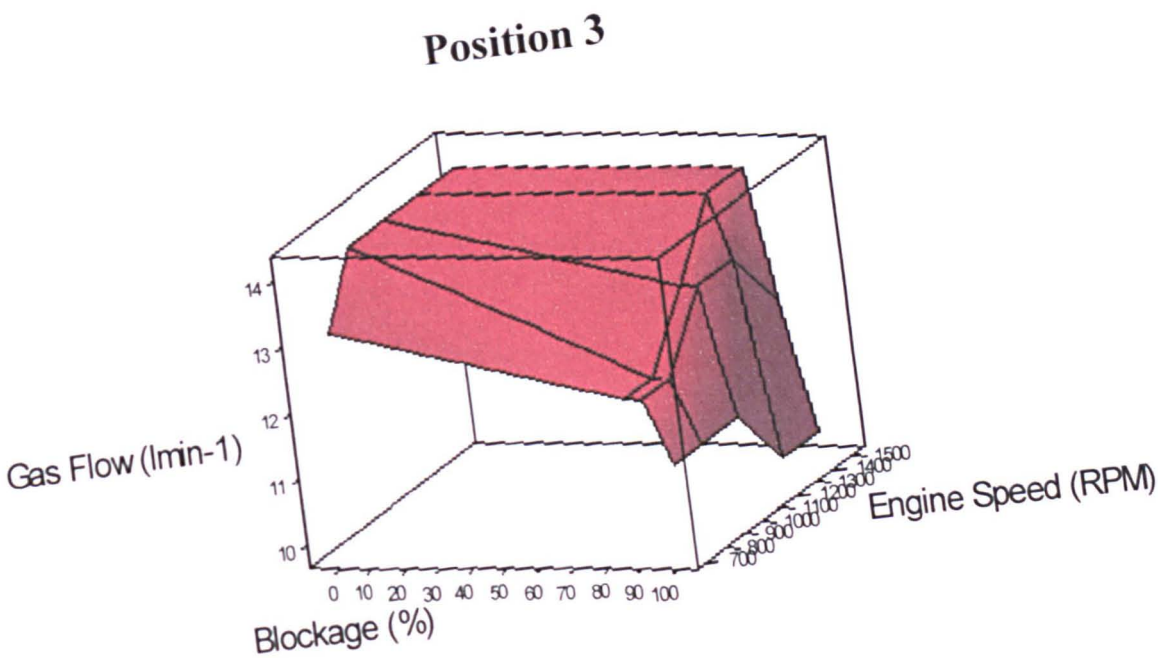
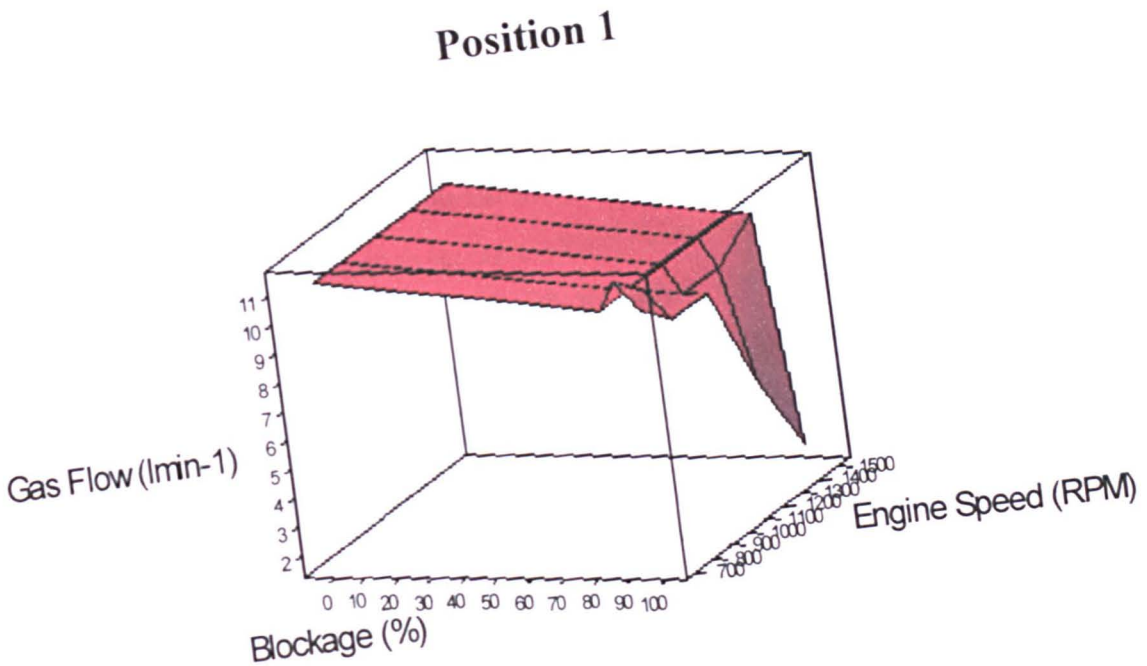


Fig 5.4a Effect of Air Filter Blockage on the Gas Flows from Sampling Position 1 and Position 3 (CAT 3406B) CONDITONS OFF LOAD



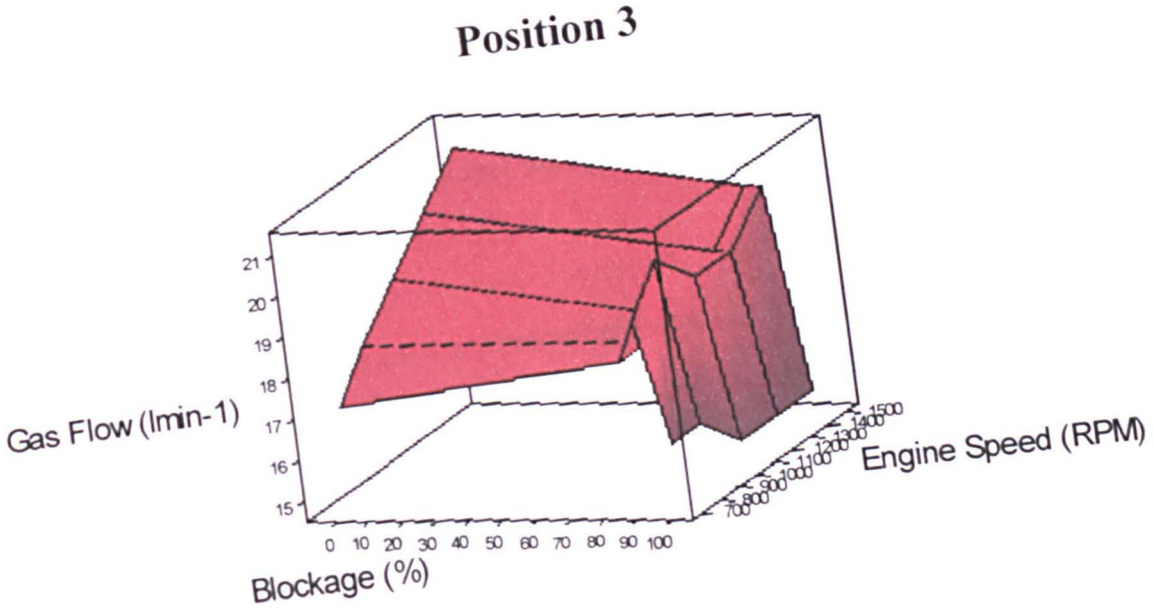


Fig 5.4b Effect of Air Filter Blockage on the Gas Flows from Sampling Position 1 and Position 3 (CAT 3406B) CONDITONS ON LOAD

The results in Fig. 5.4a and 5.4b shows that there was a large loss of gas flow at Position 1, as the filter blockage and engine speed increased. This could result in loss of oil control at the 2nd ring. Loss of oil control at the 2nd ring could have the following effect.

a) The reduction in blow by gas would not force the 2nd compression ring (Position 1) against the cylinder bore wall to its usual extent. This could result in lack of oil control at the 2nd compression ring. Lubricant could, therefore, easily pass the 2nd piston ring face, and be sampled at the top compression ring (Position 3) in increased quantities.
b) An increased level of lubricant would be expected to be in the piston ring pack if oil control was lost at the 2nd piston ring.

It should be noted that the air filter measurement system gauge on the 3406B was deemed unsuitable when the engine was used under conditions of low speed and off-load, because when the air filter was blocked the gauge did not indicate a filter blockage problem. Only when the engine was subjected to load did the gauge show that the air filter was blocked. It is therefore thought that if the engine is to be run under conditions of low speed/no-load, an alternative air filter gauge should be employed.

5.4 Comparison of 1991 and 1994 US Emission Control Ring Packs (Under Engine Conditions of Low Speed and Off Load Running)

Experiments to compare the oil mass flow for the 1991 and 1994 US emission control ring packs were undertaken. The measurement of oil mass flow was the same as described in Chapter 2 Section 2.6. The oil mass flow experiments were for a duration of 10 hours, with samples taken every 15 minutes at both sampling positions 1 and 3. Before the experimentation took place each set of piston rings were placed on the sampling piston and run at 1300 RPM under load for 10 hours to allow the rings to 'bed in'.

Results from the oil mass flow experiments to compare the 1991 and 1994 rings are shown in Fig 5.5, which shows the average amount of oil obtained from position 1 and position 3 per hour. At both 750 and 900 RPM more oil was obtained from the 1994 ring pack, when compared to the 1991 ring pack. This highlights that there was more lubricant in the piston

ring pack of the 1994 rings. However at 1000, 1100,1300 RPM more oil was obtained from the 1991 ring pack compared to the 1994 rings. The difference noted in the amount of oil obtained at different engine speeds is not fully understood, however it may be related to either the erratic nature oil flow under no load conditions, or that the 1991 rings provide generally less oil control than the 1994 rings, which could be related to the lower bore wall loading obtained from the 1991 rings.

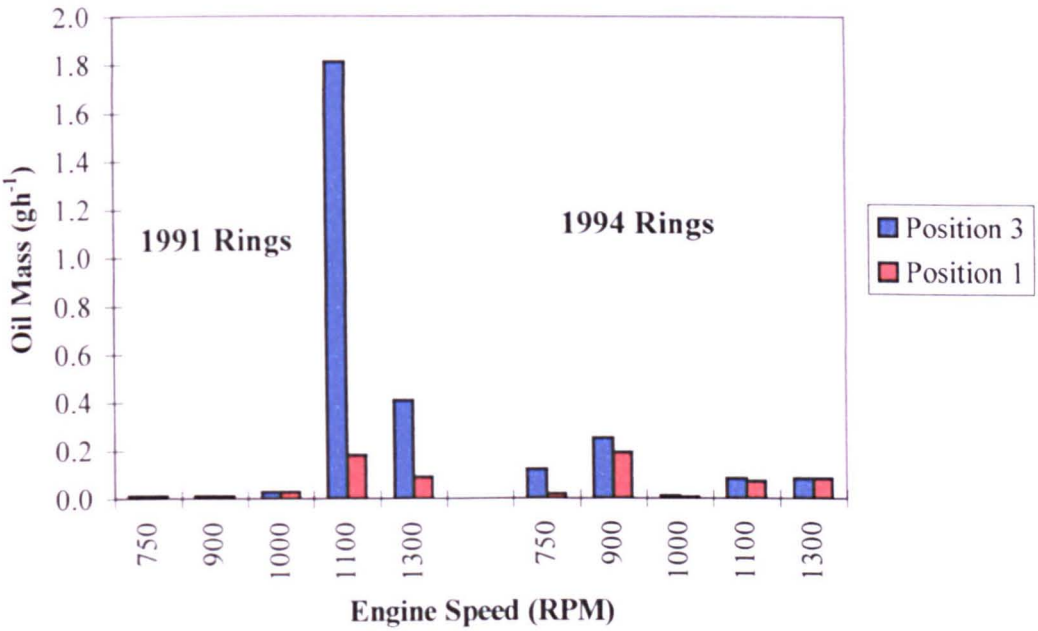


Fig 5.5 Oil Mass Flow from the CAT3406B at Various Engine Speeds using 1991 and 1994 Emission Control Piston Rings (Under No load Running Conditions)

Further investigation of the oil mass flow at an engine speed of 900 RPM is shown in Fig. 5.6. Up to 900 RPM the amount of oil obtained from the 1994 rings was considerably higher than the 1991 rings (approximately twice the amount at position 3 and sixteen times for position 1). This highlights that more lubricant was present in the 1994 ring pack compared to the 1991 ring pack. It is thought that the design of the 1994 ring pack was the reason why more oil could be present. As was stated in Section 5.1.1 the 1994 rings had a higher bore wall loading (i.e. were more tightly forced onto the cylinder bore wall) and that the width of the ring was 1mm less than the 1991 rings. Therefore there would be an increased gap between the piston ring and the piston ring groove. More oil could therefore collect in the interface between the piston and the piston ring. Because the sampling

positions were in the piston ring groove, if more oil was present in this area an increase in sample size would be detected. This is thought to be the probable reason why more oil was sampled from the1994 piston rings in some circumstances.

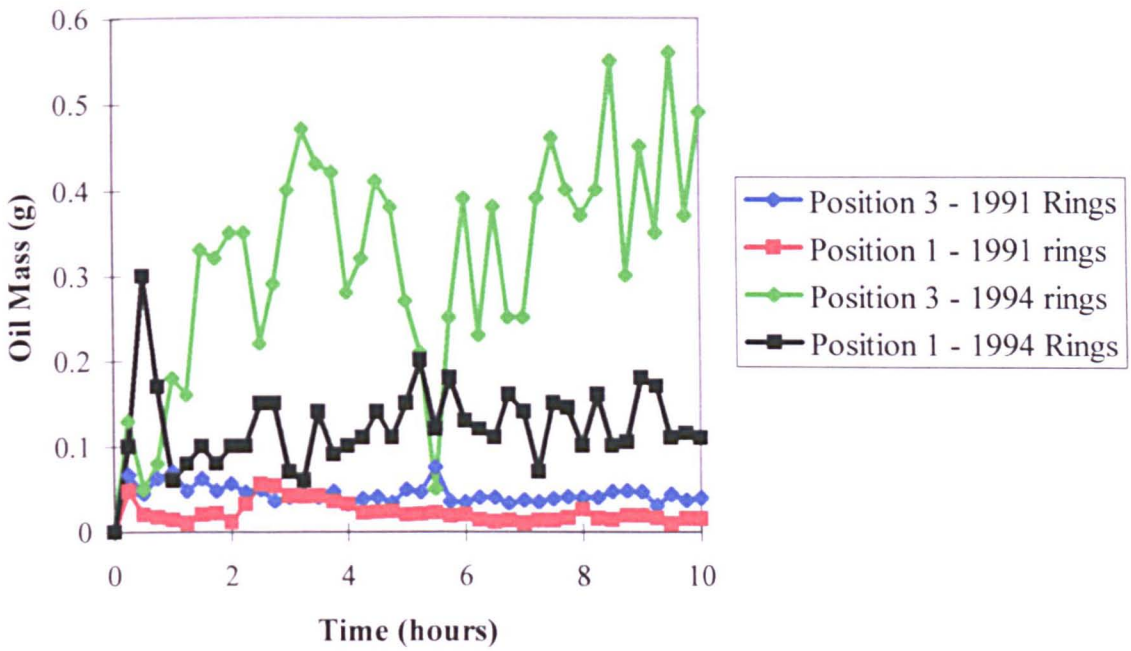


Fig 5.6 Comparison of Oil Mass Flow over Time for the 1991 and 1994 Emission Control Piston Rings in the CAT3406B at 900 RPM (Under no load engine Condition)

At increased engine speeds (i.e. 1000 RPM and above) less oil was sampled from the 1994 rings (ref. Fig. 5.5). This appears to show that the 1994 ring pack controlled lubricant flow better as the engine speed was increased. This can possibly be explained by the increased gas flow associated with greater engine speeds creating a better sealing action of the piston ring pack in the cylinder bore. However the opposite appears to be the case for the 1991 rings, where greater speeds gave rise to greater amounts of oil in the ring pack. This phenomenon is not fully understood, but the lower bore wall loading associated with the 1991 rings compared to the 1994 rings is thought to be a major factor.

The main results from the experiments are summarised below:

- The 1991 and 1994 piston rings gave consistently more oil at Position 3 than Position 1. This is thought to be due to the 'no load' engine running conditions allowing lubricant to easily pass the face of the 2nd compression ring, and thus be sampled at the top compression ring.
- At lower engine speeds (750 and 900 RPM) more oil was sampled from the 1994 rings. At increased engine speeds (1000, 1100, 1300 RPM) more oil was sampled from the 1991 rings. The results appear to show that the 1994 rings have far better oil control when the engine is running over 1000 RPM under no-load conditions than the 1991 rings. However at lower engine speeds it appears the opposite is true.

To remove the possible influence of the sampling system influencing the oil mass flow, sampling was undertaken just at position 4 (base of crown land) with the other sampling positions blocked off. This experiment gave an indication of how much lubricant would pass into the combustion chamber. The results can be seen in Fig 5.7.

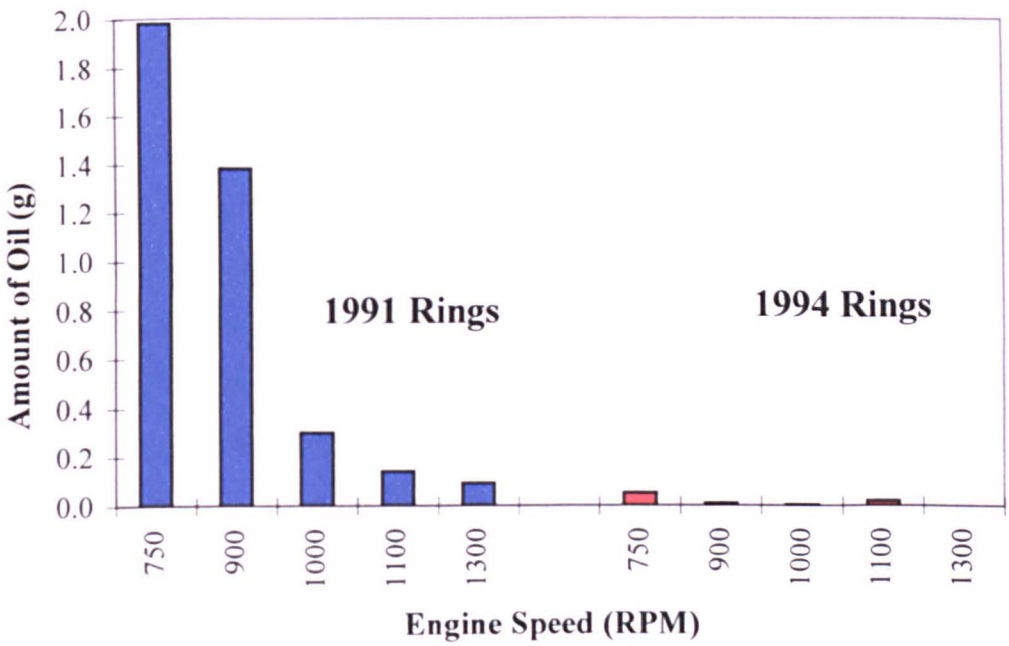


Fig. 5.7 Comparison of Oil Mass Flow at Sampling Position 4 for the 1991 and 1994 Emission Control Piston Rings at Various Engine Speed in the CAT3406B (Under no load conditions)

The results show expected results in that as the engine speed increases the oil mass flow decreases. This reduction in oil is thought to be due to:-

1. the increase in gas flow associated with increased engine speeds forcing the lubricant back down through the ring pack
2. the increase in engine temperature (associated with increased engine speed) causing the expansion of the piston in the cylinder bore therefore inhibiting oil flow into the ring pack.

It can be clearly seen that more lubricant was sampled with the 1991 rings, when compared to the 1994 rings. This highlights the superiority of the 1994 rings in reducing lubricant related emissions, by considerably reducing the amount of oil that will pass the top piston ring, and hence reducing the amount of oil that will pass into the combustion chamber. It can be seen that the 1991 rings are less efficient at controlling oil flow, by allowing more oil to pass the top ring in comparison to the 1994 rings. At an engine speed of 750 RPM approximately 1.9g less oil per hour was obtained at position 4 for 1994 rings compared to 1991 rings.

5.5 Examination into Oil Mass Appearance as Peaks

As can be seen from Fig 5.6 the oil mass flow was not constant from either sampling position or either set of piston rings. This was thought to be due to:

1. Recycling of lubricant in the piston ring zone, as described in Chapter 3 Section 3.3.
2. Variation in engine temperature, caused by fluctuations in the cooling water temperature to the engine.

An experiment was conducted to assess the phenomenon of fluctuations in cooling water temperature effecting engine temperature. Thermocouples were placed at TDC and BDC in the cylinder bore, via drilled holes in the wet cylinder liner. The thermocouples were placed 'flush' with the cylinder bore wall. The temperatures monitored during a typical engine runs at differing engine speeds are shown in Fig 5.8.

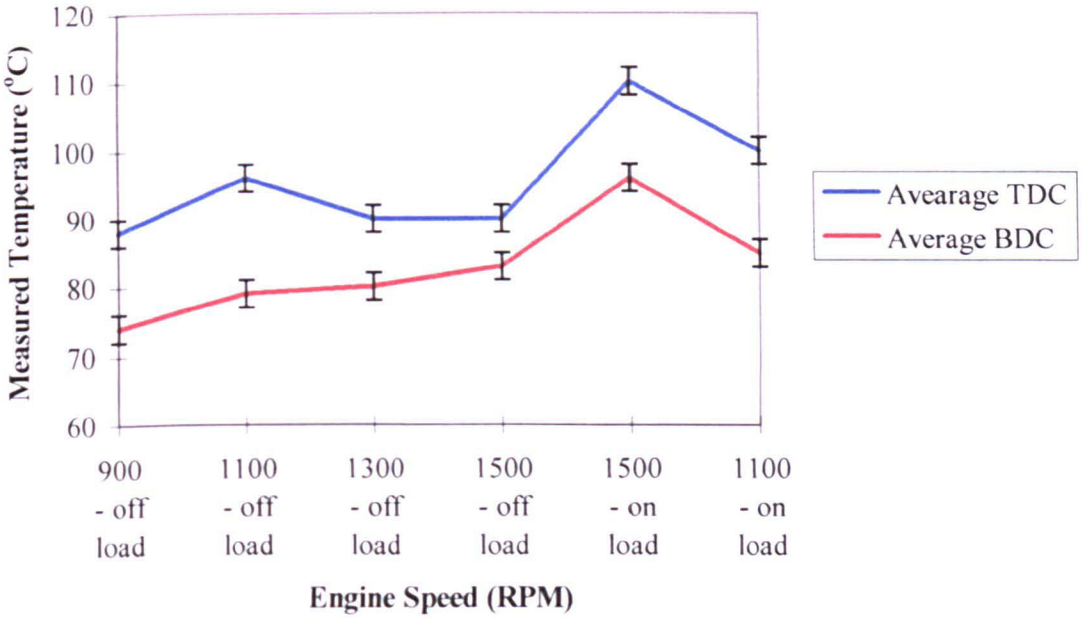


Fig. 5.8 Temperature Measured at TDC and BDC in the CAT3406B at Various Engine Speeds, under Off and On Load Conditions.

It is clearly demonstrated that there was a variation in the measured temperature at both TDC and BDC at differing engine speeds, and whether the engine was run on or off load. There is a general trend in the temperature measured, which shows that as the engine speed increased the temperature at TDC and BDC increased. Also there is a noticeable change in the measured temperature at TDC and BDC when the engine was placed under load. It was also found that the temperature during an engine run could also fluctuate by up to 2°C. It is possible that these temperature variations during an engine run could lead to differential rates of expansion/contraction of the piston in the cylinder bore, due to the piston being made of aluminium, with a steel insert for the piston rings, and the cylinder bore being made of steel. Therefore differing amounts of lubricant could enter the piston ring zone by either decreasing or increasing the gap between the piston, the piston ring and the cylinder bore (i.e. expansion of the piston would equate to a tighter oil seal). It can be argued that the measurement of temperature on the cylinder bore wall would only measure large changes in temperatures. The actual change in temperature of the piston may be more extreme and variable.

5.6 Magnesium Tracer Experiments Using the 1991 and 1994 Emission Control Piston Rings in the CAT 3406B

Magnesium tracer experiments, using oil F, were carried out to monitor oil transport to sampling position 1 and position 3 when fitted with the 1991 and 1994 US Emission control ring packs. These experiments were conducted to see if the piston ring pack design influenced the flow of lubricant through the ring pack. Experiments were carried out using the methods described in Chapter 2, Section 2.11. Injection of the oil and Magnesium sulphonate mix was by technique 1, described in Chapter 2, Section 2.11.4.

Fig 5.9 shows results for oil transport to position 1 and 3 for the 1991 and 1994 rings. As with previous experiments, to allow the engine to reach operating temperature, the engine was ran for 30 minutes prior to the Magnesium/oil mix was added to the engine. The results were evaluated in the same way as described in Chapter 3 and Chapter 2, Section 2.11, i.e. the time to reach the first maximum magnesium peak was termed the resident time. Examination of Fig. 5.9 showed that the Magnesium appeared as a plug, termed 'plug flow'. This has been discussed at length in Chapter 3, Section 3.3, and so will not be further discussed here, except to say that this was thought to be due to recycling of lubricant within the piston ring pack.

The results show that the resident time to position 1 for both the 1991 and 1994 piston rings were the same at 6 minutes. This corresponds very closely with the 8 minutes obtained for the CAT oil and 6 minutes for oil F obtained with the pre-1991 ring as reported in Chapter 3. This indicates that the oil flow to position 1 could be independent of piston ring pack design. However it should be noted that the design of the 2nd ring does not appeared to have significantly changed between the pre- 1991, 1991 and 1994 rings, therefore it is perhaps not unexpected that the oil flow has not changed between the three sets of piston rings.

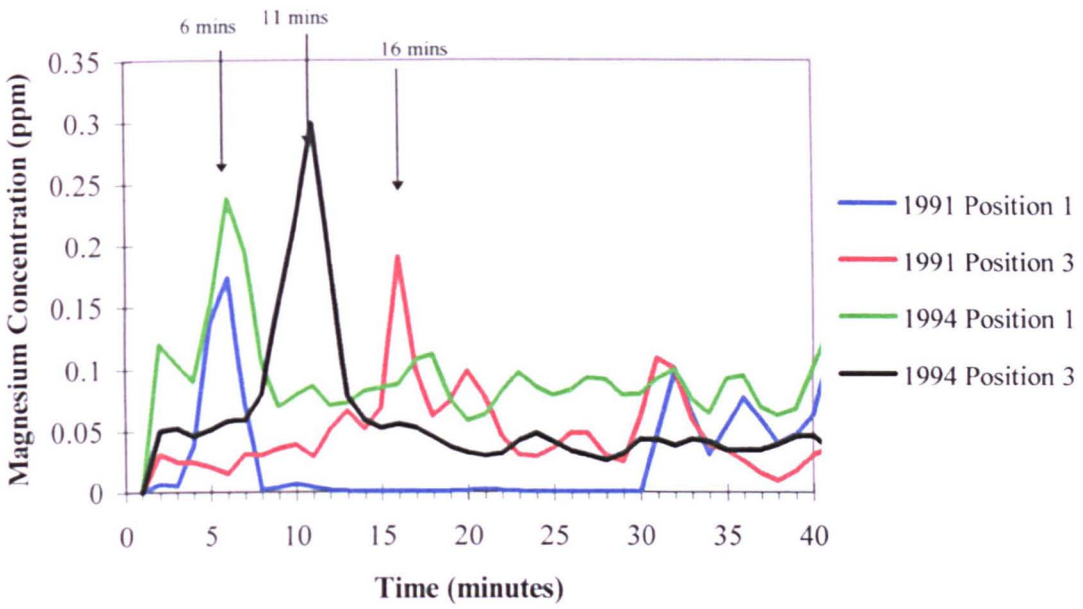


Fig 5.9 Oil Transport (Resident times) to Sampling Positions 1 and 3 for the 1991 and 1994 Emission Control Piston Rings in the CAT3406B (Speed 1500 RPM, Load 130 BHP)

The results from sampling position 3 show differences between the ring packs. The resident time using the pre-1991 US Emission Control ring pack was 30 minutes for CAT oil and 26 minutes for oil F (ref: Chapter 2 Section 2.11.8 and Chapter 3 Section 3.2). From Fig 5.9 it can be seen that the resident time was 16 minutes for the 1991 US Emission Control ring pack and 11 minutes for the 1994 US Emission Control ring pack, which showed a significant decrease when compared to the pre-1991 US Emission Control ring pack.

The resident time, and therefore oil transport, differences to sampling position 3 between the pre-1991,1991 and 1994 US Emission Control ring packs could be attributed to a higher bore wall loading effect. It has been found that the 'bore wall loading' was 1994 rings > 1991 rings > pre-1991 rings, in respect to the top piston ring. A greater bore wall loading means the top piston ring is forced against the bore wall to a greater extent, which would effectively increase the gap between the piston and the piston ring (especially if the piston ring was 'thinner' as is the case with the 1994 rings). An increase in the gap between the piston and the piston ring could lead to oil reaching the top piston ring groove at an increased rate. Assuming this theory is correct, then it would be anticipated that the oil

transport time to sample position 3 would be in the ranking of 1994 rings < 1991 rings < pre-1991 rings . Examination of the actual oil transport times shows this to be the case as the transport times were 11 minutes (1994 rings), 16 minutes (1991 rings), 26 minutes (pre-1991 rings), for experiments carried out using oil F.

Possible consequences of 'new' piston ring packs allowing lubricant to enter the piston ring zone at a quicker rate than was noted with 'older' piston ring packs are:-

- reducing the wear of the piston rings and cylinder bore.
- with lubricant entering the piston ring zone at a quicker rate the overall rate of oil degradation could decrease, which has been demonstrated (ref: Chapter 4, Section 4.8.4) where the 1994 rings showed less change in relative volatility compared to the 1991 rings.

The results show that there was very little difference noted with oil reaching position 1 with the different ring packs. A decreased oil transport time to reach position 3 has been noticed as the ring packs have become more modern. This is potentially due to the more modern rings being 'thinner' and having a higher bore wall loading, thus allowing lubricant to reach position 3 at a faster rate.

It is considered a reasonable solution to possible wear problems by allowing lubricant to enter the piston ring zone at a greater rate. However, the lubricant must not be allowed to enter the combustion chamber, as this would cause increased lubricant emissions.

5.7 Estimation of Lubricant Flow from Sampling Position 1 to Sampling Position 3 for pre-1991, 1991 and 1994 US Emission Control Ring Packs

Using the lubricant flow model described in Chapter 3 Section 3.5, an estimation of lubricant flow from sampling position 1 to sampling position 3 has been gained for the three piston ring packs. Table 5.1 details the findings.

Oil Type	Piston Ring Pack	Estimated Proportion of Lubricant Transported from Position 1 to Position 3
CAT Oil	Pre-1991 US Emission Control	51%
Oil F	Pre-1991 US Emission Control	44%
Oil F	1991 US Emission Control	72%
Oil F	1994 US Emission Control	105%*

* the level being above 100% is thought to be due to errors associated with the Magnesium measurement technique

Table 5.1 Estimated Proportion of Lubricant Transported from Sampling Position 1 to Sampling Position 3 using Different Piston Ring Packs

Table 5.1 demonstrates that there has been a considerable increase in the proportion of lubricant being transferred from sampling position 1 to sampling position 3 when comparing the pre-1991, 1991 and 1994 US Emission Control ring packs, with the proportion of lubricant increasing from 44% (pre-1991 rings) to effectively 100% (1994 rings). These results highlight that more recent piston ring pack designs are allowing more lubricant to transport from the second compression ring to the top compression ring. This can explain why more lubricant was sampled from position 3 than position 1 during the lubricant flow experiments (ref: Chapter Section 5.4) and also why less change in relative volatility was noted for the 1994 rings compared to the 1991 rings (ref: Chapter 4, Section 4.8.4).

Although it is not fully understood why the piston ring packs have been designed to allow a higher proportion on lubricant to travel from position1 to position 3, the potential benefits would be reduced wear in the piston ring-cylinder bore interface and a reduction in lubricant degradation. It should be noted, however, that although the 1994 rings have a higher proportion of lubricant transported to them this is not leading to more lubricant being transported to sampling position 4 (ref: Fig. 5.7), therefore the lubricant emissions from this ring pack would be expected to be less than the 1991 rings.

5.8 Summary of Results from Comparison of 1991 and 1994 Emission Control Piston ring Packs

The results comparing the 1991 and 1994 US emission control ring packs are summarised below:-

1. The top piston rings in the 1991 and 1994 ring packs are 'keystone' designed. The 2nd compression ring is stepped and the oil control ring is 'double knife edged'.
2. The 1994 top ring was harder to compress into the cylinder bore than the 1991 rings. Little difference were noted with the 2nd ring and the oil control ring.
3. Blocking of the air intake filter influences the amount of oil in the piston ring pack, i.e. a blocked or partially blocked air filter gives an increase of oil in the ring pack. This is thought to be due to loss of ring pressure behind the 2nd compression ring.
4. Under running conditions of low speed and no load more oil was consistently obtained from the top compression ring with respect to the 2nd compression ring.
5. At lower engine speeds (i.e. 750 to 1000 RPM) more oil was obtained from the 1994 rings than the 1991 rings.
6. Sample position 4 (base of the crown land) showed significantly less oil being present from the 1994 rings compared to the 1991 rings. This highlights that there is much better control of oil related emissions from the 1994 ring pack than the 1991 ring pack.
7. The 1994 rings had a higher bore wall loading and were 'thinner' than the 1991 rings. It is thought that blow-by gases would therefore be able to access the piston ring groove of the 1994 rings more readily than the 1991 rings. These increase levels of blow-by gases would inhibit the flow of oil into the combustion chamber, which could account for the results found in point 6, however the 1994 rings allow more oil to enter the piston ring pack.
8. Magnesium tracer experiments for sample position 1 (2nd compression ring) showed that the oil transport time was similar (between 6 to 8 minutes) for the pre-1991 , 1991 and 1994 ring packs. This is thought to be due to the ring packs being essentially the same design.
9. Magnesium tracer experiments for sample position 3 (top compression ring) showed that the oil transport time was 1994 rings < 1991 rings < pre-1991 rings (11 minutes for the

199r rings, 16 minutes for the 1991 rings and 26 to 30 minutes for the 1991 rings). One possible explanation for this is the bore wall loading being in the order of 1994 rings > 1991 rings > pre-1991 rings.

10. Comparison of the proportion of lubricant being transported from sampling position 1 to sampling position 3 highlights that the order of transport if 1994 rings > 1991 rings > pre-1991 rings. This could account for lower level of oil degradation seen with the 1994 rings and the higher level of oil noted in the 1994 piston ring pack.

CHAPTER 6

Conclusions and Recommendations

CHAPTER 6 Conclusions and Recommendations

This Chapter presents the conclusions from this study and recommendations for further work.

6.1 Conclusions

The conclusions from this work have been related to the project aims and objectives detailed in Chapter 1, Section 1.2.

The Caterpillar 3406B engine was successfully installed and modified to undertake piston ring zone sampling at four sampling positions on the piston, namely the 2nd compression ring, the accumulator groove, the 1st compression ring and at the base of the crown land. The total distance covered by the four sampling positions on the piston was 15mm.

During the course of the project the piston ring zone sampling system developed from a flexible tube - single sampling system, to a modified flexible tube - single sampling system, and finally to a dual sampling system.

The flexible tube - single sampling system was proven at speeds ranging from 750 RPM to 900 RPM, but the reliability of the system was questionable. Due to the nature of the sampling system a full engine strip down was necessary to replace the sampling system.

The modified flexible tube - single sampling system consisted of splitting the system into two parts at the support bracket, and stiffening the tube exit from the bracket connector, which were both areas of weakness with the flexible tube - single sampling system. This sampling system consistently achieved running times of 8 to 10 hours, up to engine speeds of 1500 RPM. However, the system experienced a completed failure at 1600 RPM, indicating that the system had reached its design limit.

The duel sampling system was developed to take lubricant samples from two positions simultaneously. The design eliminated, as far as possible, the use of flexible tubing. This

developed mechanical system gave a reliable and consistent performance totalling over 650 hours, at all operating conditions required. The use of the duel sampling system had many advantages, including reliability, direct comparison of lubricant degradation and gas flows from two points on the Caterpillar piston simultaneously, and the ability to measure lubricant flow within the ring pack with a greater degree of confidence. The use of the duel sampling system has demonstrated that this sampling method can be reliable, and therefore demonstrates that piston ring zone sampling could become a viable option for lubricant development and an aid to piston ring pack design.

The measurement of oil mass and gas flow was conducted using pre-1991, 1991 and 1994 US Emission control piston ring packs. A study using the pre-1991 US Emission control piston ring pack demonstrated that oil mass generally decreased as the sampling position moved nearer the piston crown, whilst the gas flow increased. A further more comprehensive study measured oil mass and gas flow at various engine speeds and loads, from the 1st and 2nd compression rings. This study demonstrated that up to 30 times more lubricant was sampled from the 2nd compression ring than at the 1st compression ring, whilst the gas flow was up to 3 times greater at the 1st compression ring than the 2nd compression ring. The oil mass from the 2nd compression ring demonstrated that as the engine speed and load increased the measured oil mass also increased. The oil mass from the 1st compression ring showed a complex trend, with 'peaks' at various engine speeds/loads, which could be a function of the engine load/speed characteristics and/or a function of the piston ring pack. The measured gas flow from the 2nd compression ring showed that a plateau was achieved between 1100 RPM and 1500 RPM. The measured gas flow from the 1st compression ring showed a similar plateau between 1100 to 1500 RPM, however distinct 'peaks' in gas flow were noted, which could also be a function of the engine load/speed characteristics and/or a function of the piston ring pack. These findings highlight the complex nature of the lubricant/gas flow regime in an internal combustion engine.

The 1991 and 1994 US Emission control piston ring packs were used to investigate the effect of slobbering, where liquid lubricant is emitted from the exhaust when an engine is run under conditions of low speed/low load for extended periods i.e. when an engine is used as a standby generator. The physical differences between the ring packs were confined to

the 1st compression ring. The 1994 piston rings were 'thinner, and harder to compress than the 1991 piston rings. It is thought that these two physical differences would effectively increase the gap between the piston ring and the piston ring groove. The low speed/low load experiments showed some surprising results. More lubricant was consistently sampled from the 1st compression ring than at the 2nd compression ring, highlighting that the piston ring pack was not functioning as intended. At lower engine speeds (750 to 1000 RPM) more lubricant was sampled from the 1994 piston ring pack than from the 1991 piston ring pack. Samples taken from the base of the crown land gave significantly less lubricant from the 1994 piston ring pack, proving that the 1994 piston rings were far superior in controlling lubricant related emissions. In summary the 1994 piston ring pack allowed more lubricant to enter the ring pack than the 1991 piston ring pack, but it allowed significantly less oil to pass the 1st compression ring.

The measurement of lubricant degradation as a function of lubricant type (mineral, semi-synthetic and synthetic), engine speed, sampling position, piston ring pack type, and diesel fuel sulphur content highlighted the complexity of modern lubricants and how they degrade. The effect of sampling position showed that, as expected, the sampled lubricant was more degraded (in terms of physical and chemical changes) as the sampling position moved closer to the piston crown. This was highlighted by the measurement of the chemical degradation of the lubricant by FT-IR, where the loss of ZDDP P=S and carbonate was tracked with increases in carbonyl oxidation and nitrate ester formation respectively. However, differences were noted in the physical degradation of the lubricants with respect to whether the sampling position was on the piston face or in the piston ring groove. Generally samples taken from the piston face showed an increase in viscosity index, either a reduction or less of an increase in volatility and lower decrease in base number, when compared to samples taken from behind the piston rings. The noted differences are thought to be due to the location of the sampling position. It can be argued that the samples taken from behind the piston rings would be subjected to an environment more prone to shearing of the lubricant, due to the action of the piston ring movement in the groove, and lower metal temperatures which could allow the samples to be contaminated by diesel fuel to a greater extent. A more comprehensive analysis of the data using common statistical techniques showed that lubricant degradation was significantly affected by oil type, sampling position, fuel sulphur

content (although this only affected base number) and piston ring pack type, where it was found that the use of the 1994 piston ring pack changed lubricant volatility to a lesser extent than the 1991 piston ring pack. This project demonstrated that engine speed/load did not significantly affect lubricant degradation. In general terms the synthetic lubricants tested showed a decrease in viscosity, whereas the mineral and semi-synthetic lubricants showed an increase in viscosity. In general the synthetic lubricants also showed the least change in volatility, and the least change in base number.

The statistical evaluation demonstrated that synthetic lubricants showed a consistent reduction in viscosity, which it thought to be due to shearing of carbon chains. Any shearing will result in a viscosity decrease, due to the reduction in molecular weight. This will effect synthetic lubricants to a greater extend due to their more uniform carbon chain length. The use of Principle Component Analysis has demonstrated that lubricant samples can be classified according to their degradation level i.e. viscosity increase/decrease/no change, high/medium/low level of chemical degradation. This highlights the potential of using piston ring zone sampling as a quick screening test to assess lubricant quality.

Particle size analysis of samples from the four sampling positions demonstrated that the size of particles increased as the sampling position moved nearer to the piston crown, as would be expected due to the sampling positions being nearer to the combustion chamber. The measured particle size was generally not normally distributed, with the 2nd compression ring showing distributions centred on 1.2μm, 3μm and 5μm, whilst the 1st compression ring has distributions centred on 3μm and 7μm. The particle size distribution centred around 1μm is thought to be due to soot particulates formed during the combustion process. Particles greater than 1μm are possibly due to the agglomeration of these soot particulates. This possibly highlights an ineffective dispersant additive in the oil formulation.

The effect of diesel fuel sulphur content on the size of particles in lubricants was very noticeable. Using a 1% sulphur fuel with a mineral based lubricant gave particle size distributions centred on 1.2μm, 3μm and 5μm for the 2nd compression ring, whilst a 0.3% sulphur fuel gave distributions at 1μm and 15μm and distributions at 1μm and 200μm with a 0.05% sulphur fuel. Disturbingly, sump samples also showed a large distribution centred

at 100μm from samples run with the 0.05% sulphur fuel. Synthetic based lubricants performed better using different sulphur fuels, with distributions at 1μm and 10μm with the 0.3% sulphur fuel and 1μm and 20μm with the 0.05% sulphur fuel. It should be noted that the size of some of these particles are greater than some of the engine clearance tolerances within an internal combustion engine, and therefore highlights potential problems e.g. piston ring sticking. The size of the particles measured demonstrates that the lubricants tested did not effectively disperse the particles, which questions the effectiveness of the dispersant additives in the lubricant formulations. The need to use lower sulphur diesel fuels, due to government legislation to reduce emissions, highlights the need to produce and use lubricants with more effective dispersant additive packs, as it has been demonstrated that lowering the sulphur content in diesel fuel increases the particulate size in lubricating oils.

The measurement of lubricant transport from the sump to the various sampling positions highlighted that the lubricant transport in a modern 'low emission' diesel engine is considerably slower than 'older' engines, which we not designed to current emission regulations. Experimental work on the Caterpillar 3406B using the pre-1991, 1991 and 1994 US Emission control piston ring packs demonstrated that the lubricant took between 11 to 30 minutes to travel from the sump to the 1st compression ring. In comparison a transport time of 3 to 5 minutes was obtained using a Petter AA-1 and a Caterpillar 1Y540. Care has to be taken when directly comparing these transport times because the relative difference in the sizes of the engines would be a factor in lubricant transport time. However there is a potential that lubricant transport times have increased, which would imply that lubricants now have to perform under the same arduous conditions, with thinner oil films, for longer time periods. It can therefore be argued that these conditions are placing unacceptable stresses on mineral based lubricants and that synthetic based lubricants should now be recommended by engine manufacturers, due to them possessing inherently better viscosity and volatility characteristics. A lubricant transport model was developed by taking into account the lubricant transport times and the distance the lubricant had to travel up the piston to the various sampling positions. This model demonstrated that of the lubricant reaching the 2nd compression ring 50% of it was transported to the accumulator groove, 88% of the lubricant reaching the accumulator groove was transported to the 1st compression ring, whilst 42% of the lubricant reaching the 1st compression ring was

transported to the base of the crown land. These findings highlight that the pistons rings act as controlling factors by inhibiting oil flow. Using the model it can be shown that 19% of the lubricant that reaches the 2nd compression ring is transported to the base of the crown land. It can be argued that all of the lubricant reaching the base of the crown land could be burnt, and thus produce lubricant emissions.

Lubricant transport was also measured for the 1991 and 1994 US Emission control piston ring packs. This work demonstrated that the time taken for the lubricant to travel from the sump to the 2nd compression ring was essentially constant, and independent of ring pack design or lubricant used, with transfer times continually being measured between 6 to 8 minutes. However , the time for the lubricant to travel from the sump to the 1st compression ring was affected by ring pack design, with transfer times being 11 minutes for the 1994 piston ring pack, 16 minutes for the 1991 piston ring pack and 26 to 30 minutes for the pre-1991 piston ring pack. The changes in transfer time are believed to be due to physical differences noted in the design of the 1st compression ring i.e. the 1994 piston rings were thinner and harder to compress than the pre-1991 and 1991 piston rings. Using the developed lubricant transport model it can be demonstrated that for the pre-1991 piston ring pack 44% of the lubricant from the 2nd compression ring is transferred to the 1st compression ring, compared to 72% for the 1991 piston ring pack and essentially 100% for the 1994 piston ring pack. This highlights that more recent piston ring pack designs are allowing more lubricant from the 2nd compression ring to transfer to the 1st compression ring. This is considered to be an acceptable way to reduce the level of lubricant degradation and engine wear, however the more lubricant that is allowed into the piston ring pack the greater the probability of lubricant being transported to the combustion chamber, producing lubricant related emissions. It is therefore thought that a compromise has to be reached by allowing sufficient lubricant into the piston ring pack without inducing higher levels of lubricant related emissions. This compromise may be at the expense of increased levels of lubricant degradation, which also highlights that synthetic lubricants should now be recommended by engine manufacturers. The use of the lubricant transfer technique could be employed by piston ring manufacturers to monitor and measure lubricant flow within piston ring packs as an extension to the theoretical lubricant flow models employed. It is thought that the use of practical experiments would help in the development of piston ring packs in

order to minimise lubricant degradation, without compromising on lubricant related emissions.

6.2 Recommendations for Further Work

During the course of this project areas have been identified which could be evaluated further, to give valuable insight into lubricant performance in the piston ring zone.

- Particle size analysis on lubricant samples ran with different fuel sulphur levels has demonstrated that lower sulphur fuels produce particles of increased size. The lubricants tested during this project highlighted that the dispersant additives may not be functioning as intended as the fuel sulphur content decreases. A programme of work could therefore be set up to investigate this further by evaluating lubricants containing various dispersant additives against diesel fuels of varying sulphur content. Particle size analysis could be used to monitor the lubricant's performance, which could lead to a insight into which dispersant additives should be used with lower sulphur fuel.
- The data obtained from this project was considered to be 'noisy' i.e. repeat engine runs produced variable results in terms of lubricant degradation. Possible explanations for this is that piston ring zone sampling produces variable results, or that other variables i.e. engine build or environmental conditions, were having an effect on the piston ring zone samples. To investigate the repeatability of piston ring zone work a programme should be set up where only a limited number of lubricants are tested a number of times. The engine runs should, wherever possible, be carried out under controlled conditions i.e. at constant environmental conditions. If it is not possible to control these conditions then the conditions should be constantly monitored i.e. environmental temperatures and engine temperatures at key points on the engine. By evaluation of this information it should be possible to quantify the effect of variability associated with engine build, the variability associated with environmental conditions, and therefore the variability associated piston ring zone sampling (assuming the variability associated with the analytical test methods are previously known). This assessment and breakdown of where variability occurs would provide a valuable insight into the viability of using piston ring zone sampling as a recognised engine test method.

- The project has demonstrated the use of piston ring zone sampling as a potential quick screening method for evaluating experimental lubricants. This could be extended further by setting up a programme of work which consists of running a series of standard lubricants i.e. sequence III lubricants, or lubricants that have historically been shown to work 'well' and 'badly' in routine use, to obtain piston ring zone samples. These ring zone samples should be tested by a suite of analytical tests and analysed using Principle Component Analysis in order to produce a lubricant quality/performance chart. Experimental lubricants could thereafter be run and compared against the quality/performance chart, thus providing a very quick method to evaluate how the lubricant would perform.
- The lubricant transport work identified the potential to practically monitor lubricant transport in the piston ring pack. Further experimentation in this area could be used to refine the predictive lubricant transfer models used by industry, and to modify the models by taking into account levels of lubricant degradation.

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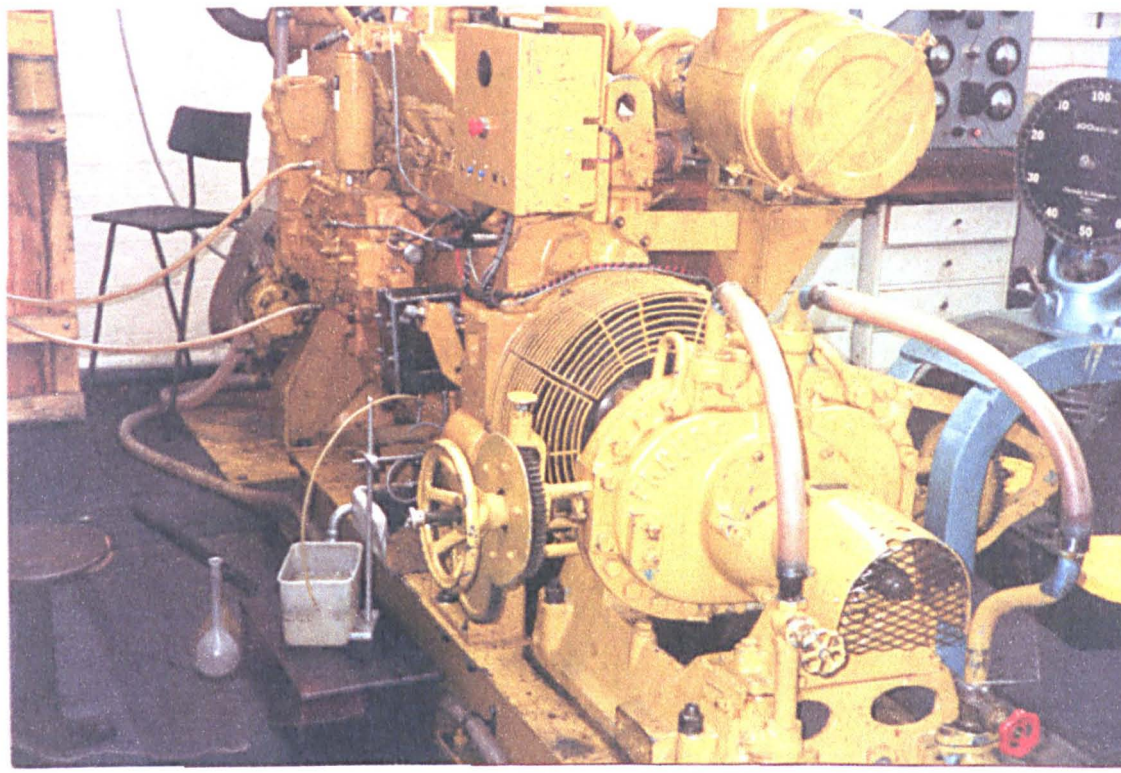
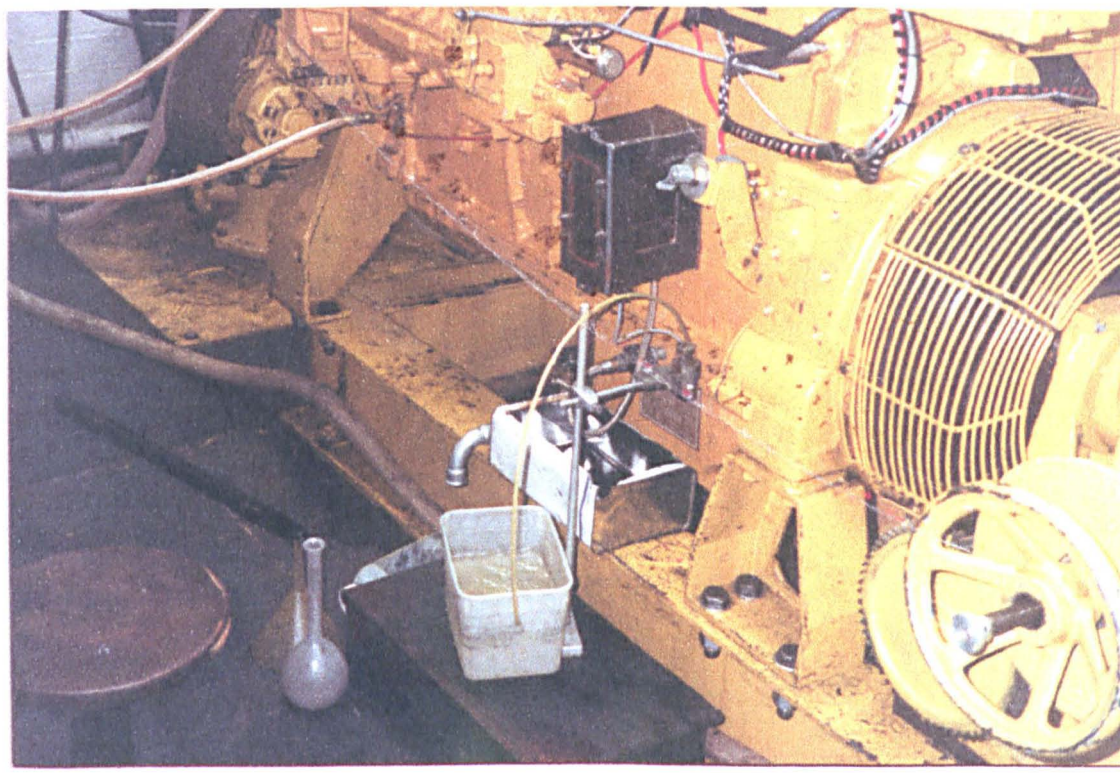
APPENDICES

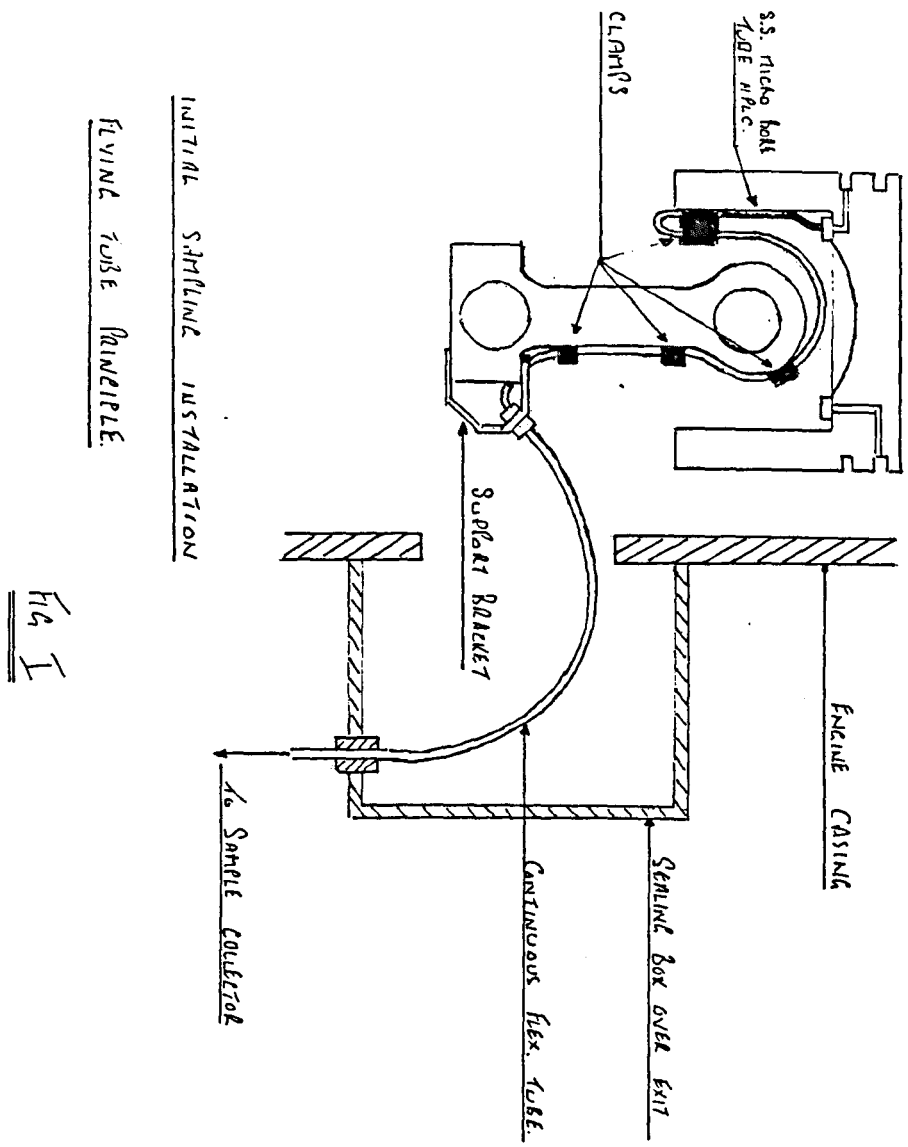
APPENDIX A





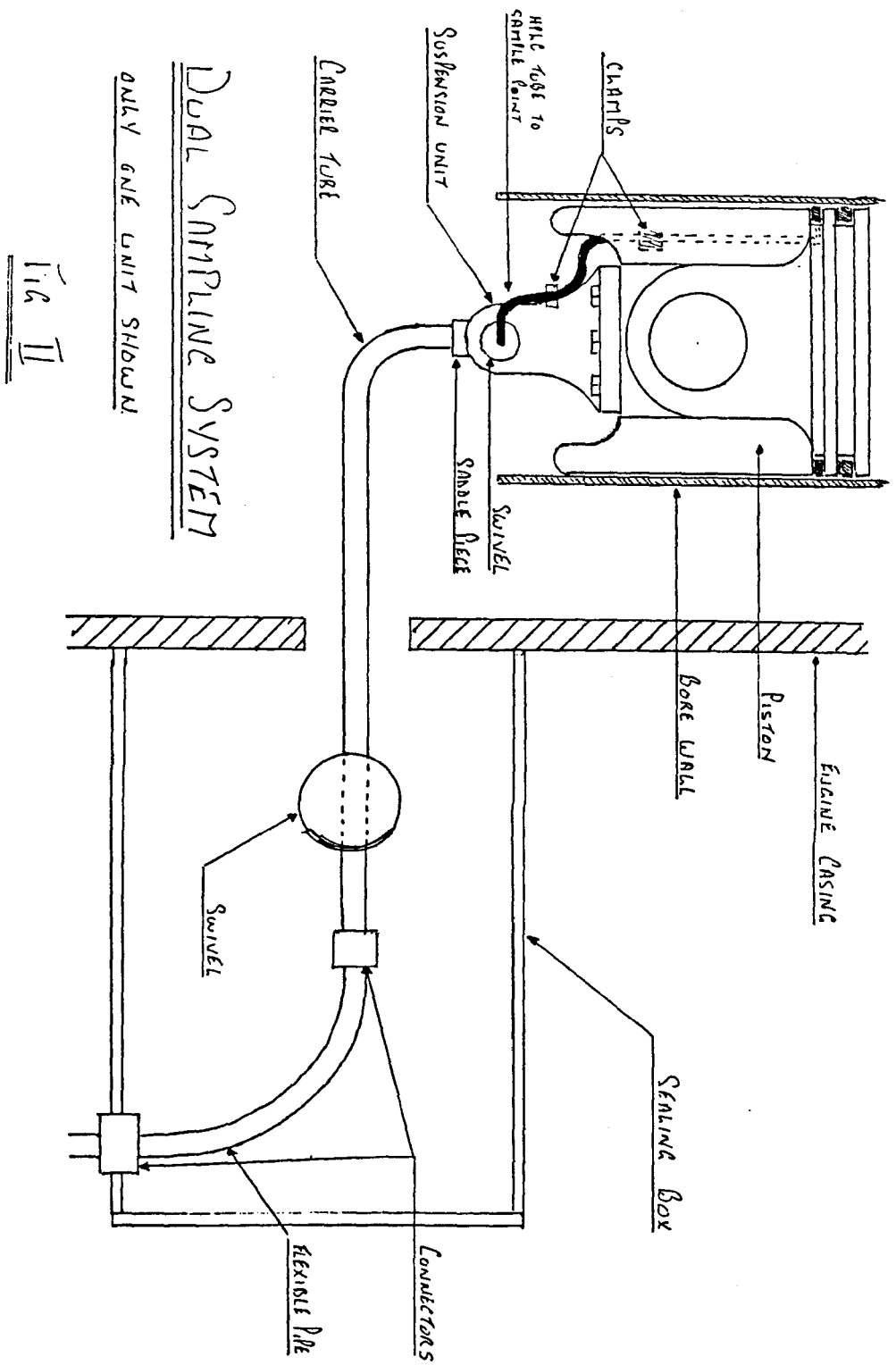
Photographs of the Caterpillar 3406B Engine

APPENDIX B



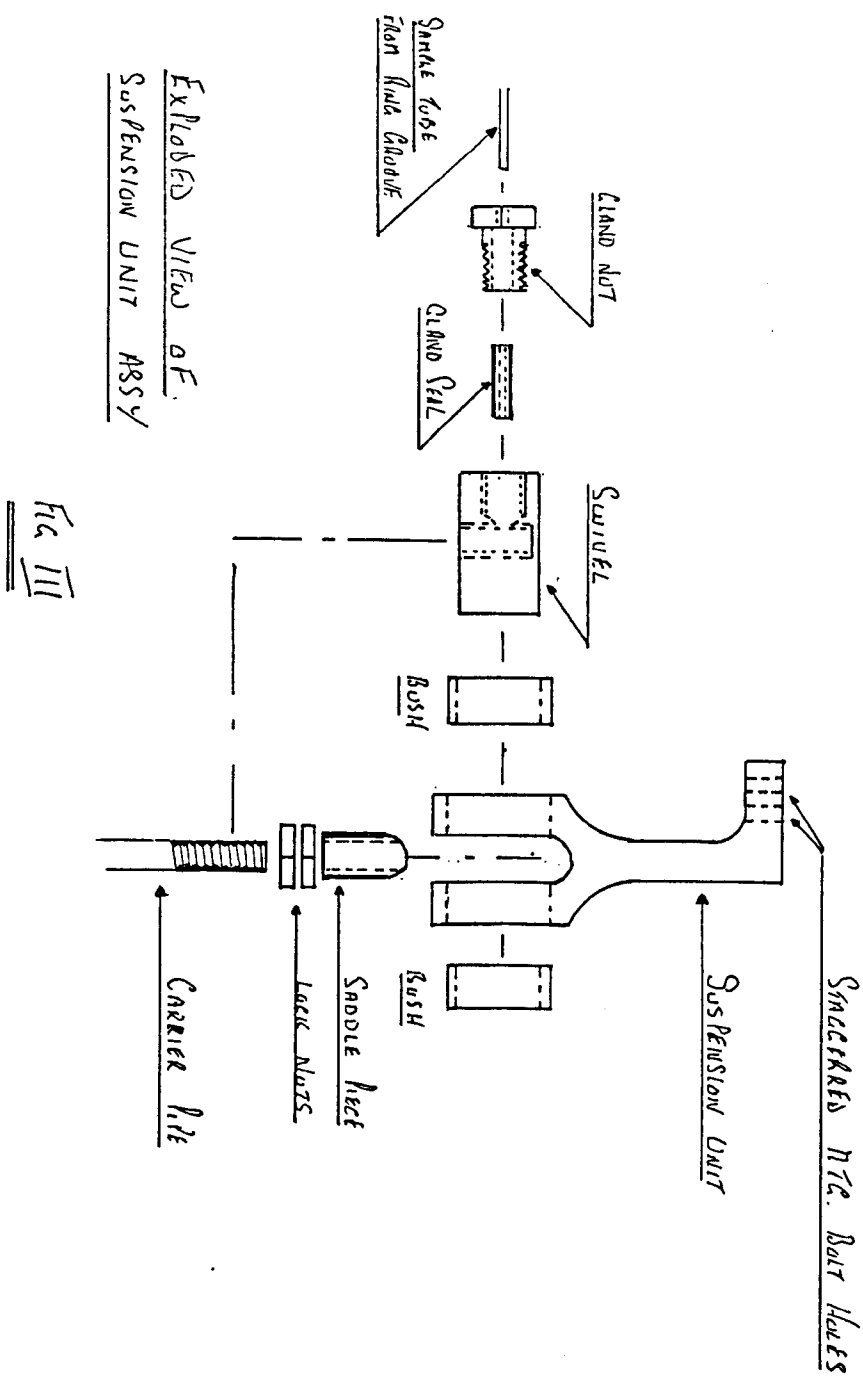
Flexible Tube - Single Sampling Position Sampling System

APPENDIX C



Duel Sampling System

APPENDIX D



EXPLODED VIEW OF SUSPENSION UNIT ASSY

FIG III

Gland Nut and Seal

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PUBLISHED PAPERS

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